VOL. LIII, NO. 8, AUGUST, 1958

THE Journal OF THE AMERICAN LEATHER CHEMISTS ASSOCIATION

PUBLISHED MONTHLY BY
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PUBLICATION OFFICE, EASTON, PA.

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The Journal

of the

AMERICAN LEATHER CHEMISTS ASSOCIATION

Vol. LIII AUGUST 1958 No. 8

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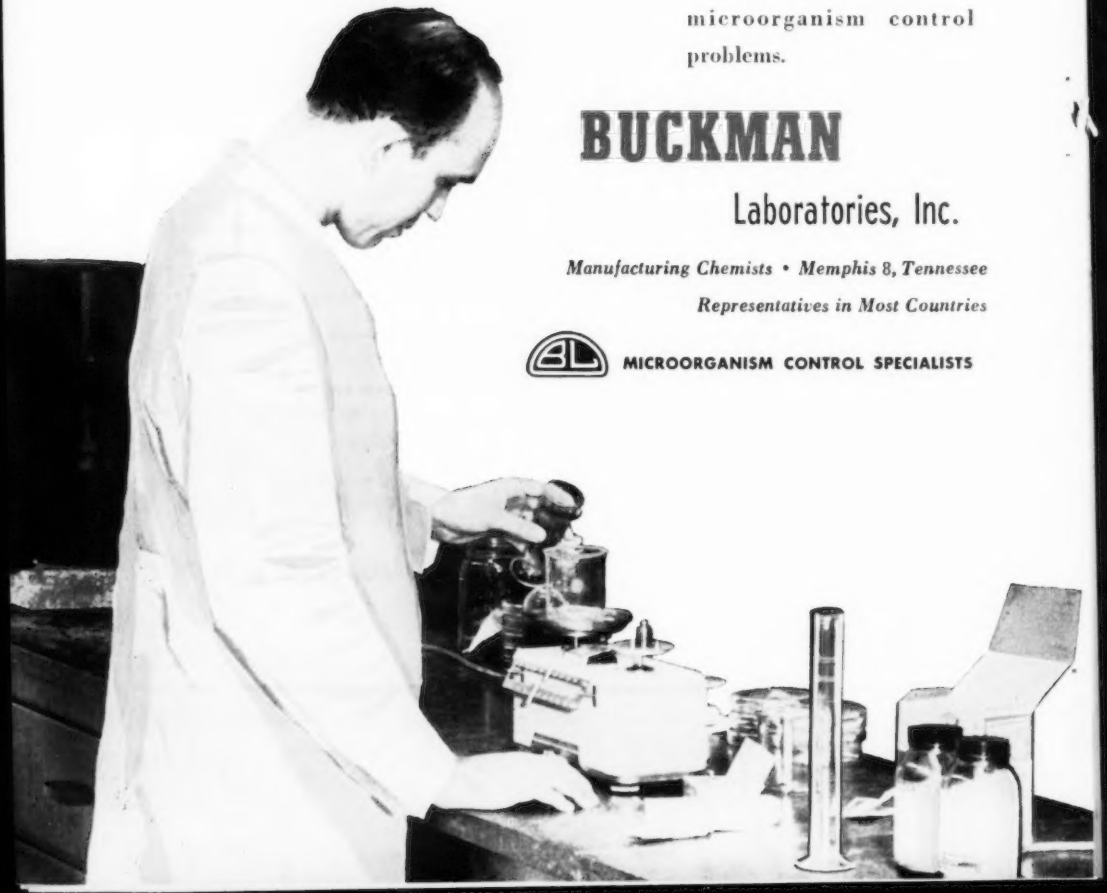

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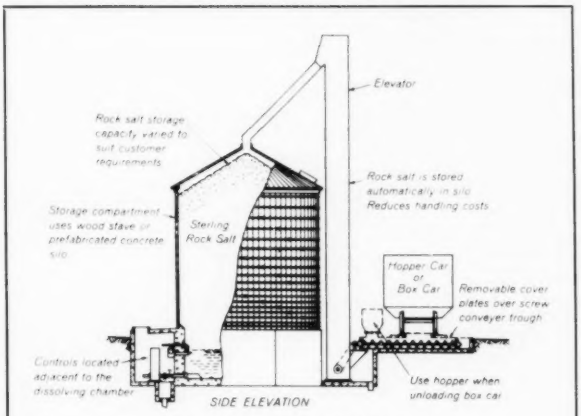
How to Store Large Tonnages of Rock Salt in Limited Space

One of the best ways to increase salt-storage capacity, without taking up valuable indoor or outdoor plant space, is to install a "Silo Lixator." Developed by International Salt Company, this space-saving unit — which *combines* salt storage with brine making — is especially practical where storage areas in or around the tannery are at a premium.


Elevator
Rock salt storage capacity varied to suit customer requirements
Rock salt is stored automatically in silo. Reduces handling costs.
Storage compartment uses wood stave or prefabricated concrete silo
Sterling Rock Salt
Hopper Car or Box Car
Removable cover plates over screw conveyer trough
Controls located adjacent to the dissolving chamber
Use hopper when unloading box car
SIDE ELEVATION

TYPICAL SILO LIXATOR

The Silo Lixator makes excellent use of limited storage space, because it stores rock salt in a high, vertical column. The upper portion of the column stores dry salt. The bottom of the salt column rests in a dissolving tank, where water is added to make brine. Whenever salt in this tank is dissolved to make brine, it is immediately replaced by dry salt flowing down from the upper portion of the column.

In addition — and this is an important, money-saving feature — there is ample space in the dissolving tank for brine storage. (The brine occupies the voids between the salt crystals.) This method of storing salt — with both dry salt and saturated brine present — is one form of wet storage. It does away with the need for a brine-storage tank.

For storing any given amount of rock salt in dry form, ample space can be provided in the silo portion, by building it to the proper height. The rugged steel, wood-stave, or prefabricated-concrete construction of the silo assures long, dependable service.

Rehandling eliminated. Because dry salt stored in the Silo Lixator is dissolved to make brine, there is never any salt rehandling in the tannery. A simple piping system, installed where it won't interfere with other operations, carries this brine to points of use, where it's available simply at the turn of a valve. What's more, every drop of Lixate Brine is completely self-filtered by the rock salt crystals at the bottom of the dissolving tank. Result: 100%-saturated brine that is always pure and crystal-clear.

Incoming salt shipments need no special handling. One of the more common delivery methods is the one pictured above: rock salt flows directly from a hopper car into a screw-conveyor trough, which, in turn, carries the salt to a bucket elevator. Another unloading method, possible when the elevator boot is low enough to permit gravity feed from the hopper car, is the gravity-feed chute.

For more information on salt storage . . . and on modern techniques for handling and using salt or brine . . . contact International Salt Company. One of our experienced sales engineers can show you how the Lixate Principle is benefiting salt-using companies everywhere. He can also help you select the brine-making set-up that best suits your specific needs. Ask him . . . send a card or letter to International Salt Company, Inc., Scranton 2, Pa. . . . or contact our nearest sales office.

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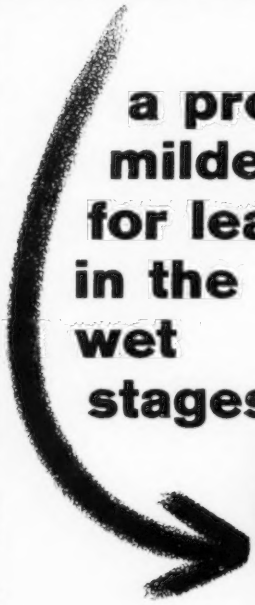

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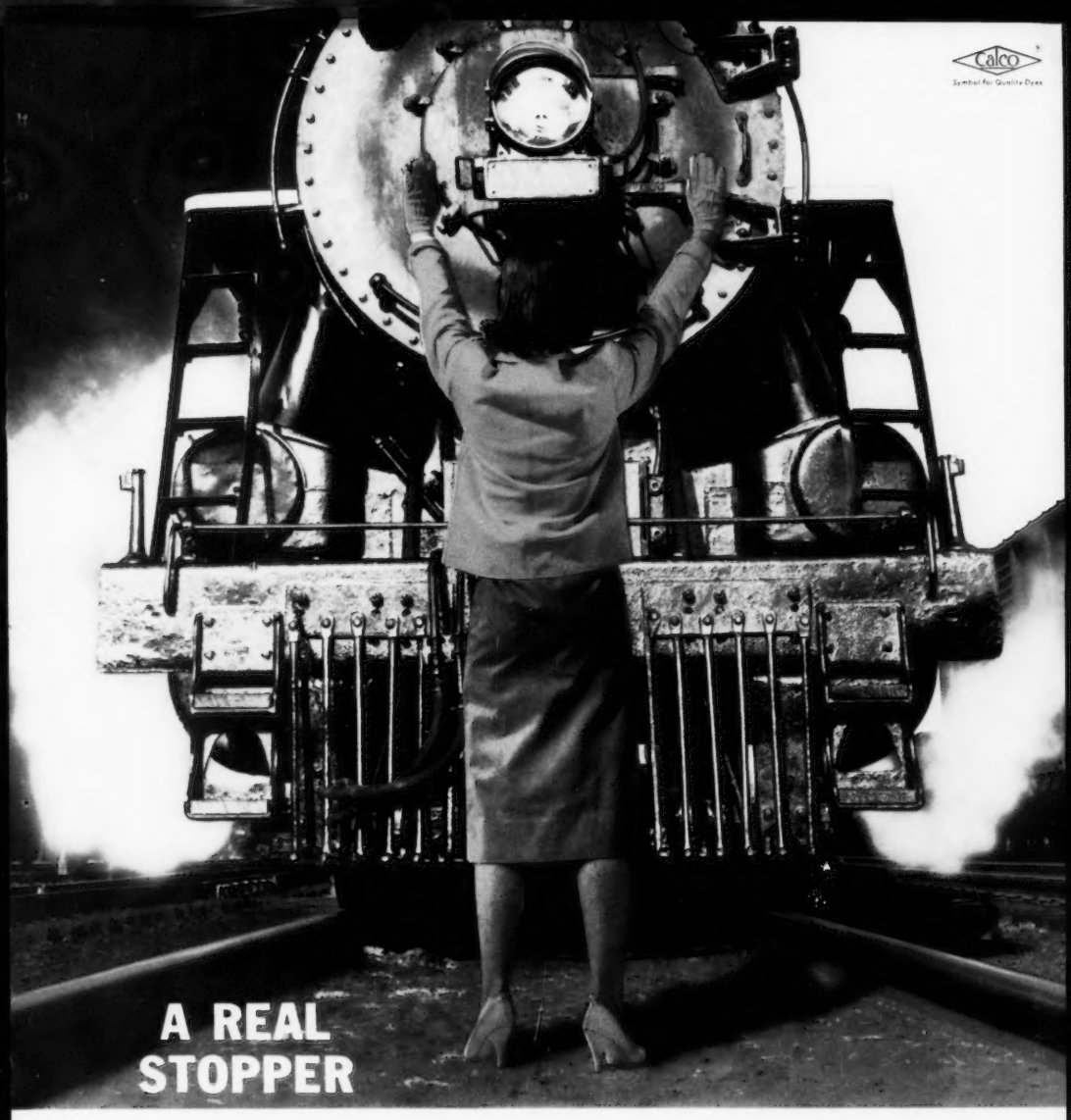

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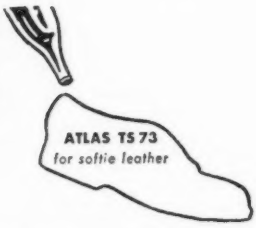

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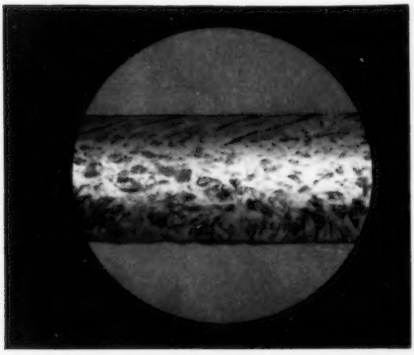

Cross-section photomicrograph illustrates controlled penetration of Mobiltan 275. Special qualities permit surface nourishment and enough penetration to give the desired mellowness.

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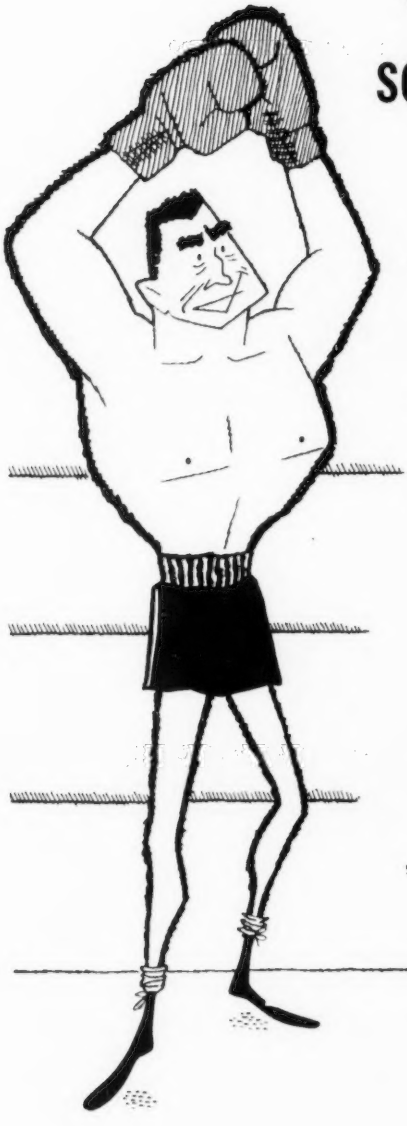

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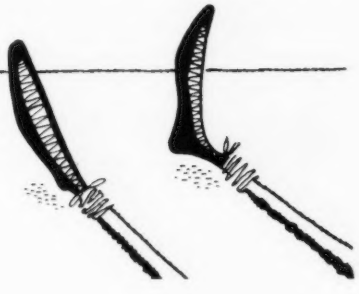

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WATER RESISTANCE OF SHOE UPPER LEATHERS TREATED WITH COMPOSITIONS OF STEROLS AND OF HYDROCARBON SULFONATES OF HIGH MOLECULAR WEIGHT

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ABSTRACT

Two types of compositions are described which give moderate to high water resistance to chrome and retan upper leathers when tested on a dynamic machine. These compositions are as follows:

(*a*) Free sterols and sterol-like bodies derived from wool grease and incorporated into the leather with petroleum oils, petrolatum, and fatty acids.

(*b*) Hydrocarbon monosulfonate salts of high molecular weight with fatty acids, fatty alcohols, or alkyl phenols. The sulfonate salt, usually that of sodium and of molecular weight preferably 500–700, may be derived by purification of a by-product from petroleum refining or may be synthesized.

Tests on leathers treated with these compounds indicate that they impart somewhat less water resistance than two other commercial water repellent agents, although the order of magnitude of the water resistance is about the same. The sulfonate composition is superior in performance to the sterol and also lower in cost.

Leather treated with the compositions must have about one-third of the free pore space filled to give optimum results. The leather obtained by these treatments resembles conventional stuffed upper leather with respect to feel and has similar breathing qualities. Treated leathers show some within-lot variations attributed to unknown factors in hide and skins, but a moderately high water resistance may be obtained on an average with suitably tanned and finished leather.

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INTRODUCTION

The development of the water-resistant treatments described in this paper was initiated before World War II, using separated and fractionated components of wool grease or degras. Glove, upper, and sole leathers treated

with compositions of these materials showed improved water resistance by static tests, and the leathers possessed good qualities of feel, and freedom from greasiness.

With the advent of dynamic testing methods (1, 2) and the reported failure by these methods of all of the conventional water-resistant treatments used at that time, a reappraisal of wool grease components was made. The characteristic of these materials to absorb and isolate water suggested a possible means of nullifying water penetration caused by the pumping action of the tiny pores in the leather.

To facilitate the screening of the compositions a dynamic test apparatus was devised which is described later in this paper. Some compositions were found to give high dynamic test results, and further testing indicated that this was due essentially to the free sterols and sterol-like bodies. These substances are adsorbed strongly on the fibers, particularly those of chrome tannage. Many other chemicals possessing a similar type of surface-active quality were screened for possible application. Of particular promise were certain hydrocarbon sulfonates of high molecular weight. The two types of water-repellent compounds (3) described in this paper were evolved from this work.*

COMPOSITIONS

The materials tested consisted of formulations composed of the active ingredient and other less expensive compounds which were compatible and were known to have water-resistant properties.

Compositions with sterols.—The active ingredients consisted of the purified wool alcohols which were separated from wool grease by various saponification and separation processes (4). These were mixed with mineral oil or petrolatum. Microcrystalline waxes, lanolin, liquid fatty acids, or fatty alcohols may also be used. Wool grease of suitable sterol content, saponified to produce a mixture of free alcohols and fatty acids, may also be used. This mixture is used directly. The use of purified wool alcohols is, however, preferable and was used to obtain the data given in this paper. Treatment E exemplifies this type, and its composition is as follows:

Purified wool grease alcohols	30%
Petrolatum	20%
Nonstaining degras (25–30% free fatty acid containing some free sterols)	25%
Polymerized stable fatty acids of linoleic acid	25%

*Extensive application studies and evaluations of tannages and treatments were carried out under QMC Contract (1951–52) No. DA 44-109-qm-496: Development of Plant Scale Application of a Process for Dynamic Waterproofing of Retan Upper Leather.

Leather treated to contain 15% of this composition would contain 2 to 2.5% sterols or sterol-like bodies.

The alcohol portion of wool grease usually contains in the vicinity of 45% sterols and sterol-like bodies, such as cholesterol, dihydrocholesterol, oxycholesterol, and isocholesterols (4). No attempt has been made to determine which of these substances in the mixture is the most effective.

Compositions containing pure cholesterol or sitosterols derived from vegetable oils are not so effective as the mixed wool grease sterols, and it is believed that the less saturated and more liquid sterol or sterol-like bodies are the most effective.

Compositions with sulfonates.—The hydrocarbon sulfonates of high molecular weight used to improve water resistance of leathers are characterized by the general formula:

$$R\ (SO_3M)_n$$

where R is a hydrocarbon radical having about 27 or more carbon atoms, M is a metal, ammonium, or amine radical, and n is ordinarily one but may be two or more, provided there is not more than one sulfonate group to every 27 carbon atoms in the molecule.

It should be noted that the sulfur atom in the sulfonate group is combined directly with the carbon atom in the hydrocarbon chain, unlike sulfated oils (so-called sulfonated oils) and hydrocarbon sulfates derived from fatty alcohols where the sulfur atom is attached to the hydrocarbon chain through an oxygen atom. The sulfur-to-carbon linkage produces a highly stable substance which does not hydrolyze or break down in the leather.

One preferred sulfonate compound is a mono sodium sulfonate of a mixture of hydrocarbons having 30–40 carbon atoms with molecular weights of 500–700. The hydrocarbon mixture must have sufficient branched chain, cyclic, or aromatic components to be noncrystalline.

The hydrocarbon sulfonate of high molecular weight may be obtained by segregation and purification from a by-product of petroleum refining (petroleum sulfonate) or by synthesis. There are numerous satisfactory synthetic processes which include direct sulfonation of a suitable alkyl aryl hydrocarbon by sulfuric acid or sulfur trioxide, or sulfochlorination (Reed reaction) of alkyl or alkyl aryl hydrocarbons followed by hydrolysis.

To be effective, the composition must contain at least 4% by weight of the sulfonate. The sulfonate is ineffective if used alone. It functions only in the presence of one or a mixture of liquid fatty acids, polymerized fatty acids, fatty alcohols, or long chain alkyl phenols having an alkyl group of 14 or more carbon atoms. The concentrations of these materials may range from 10 to 80%. Among other favorable materials in the composition, singly or as mixtures, are mineral oil, petrolatum, microcrystalline waxes, and ozocerite wax.

The composition of a representative sulfonate formulation is as follows:

Hydrocarbon sulfonate (actual)	12%
Mineral oil	13%
Petrolatum	22%
Microcrystalline wax	5%
Stable liquid fatty acid	48%

Leather treated to contain 15% of this mixture will contain 1.8% of sulfonate.

METHODS OF TREATMENT

The compositions may be readily incorporated into the leather by conventional methods. Drum stuffing or fatliquoring is satisfactory, although trouble may be experienced in finishing. The most reliable and easiest way to apply the material is by hot swabbing or by applying solutions to the dry finished leather. Solutions in high-flash naphtha may be applied by dipping, roller-coating, brushing, padding, or spraying.

The specimens used to obtain the results shown in Fig. 2 and Table I were treated by brush applications of solutions of the various materials in petroleum naphtha and then dried free of solvent.

To obtain maximum effectiveness from the treatments, a number of precautions should be taken in the methods of tanning and finishing the leather. If the leather is fatliquored in the normal way, emulsifiers should be of the anionic or nonionic type and kept as low as possible. Anionic hydroxy-type sulfated oils such as castor should be avoided, as well as cationic-type fatliquors, oxidized fats, and materials like moellon degras and resin acids such as tall oil.

Since the materials combine to some extent with the chrome-tanned fiber, chrome leathers which are highly retanned or those which contain high amounts of masking salts or some auxiliary tanning materials are more difficult to treat effectively. Sodium formate gives less trouble than other masking salts. Some vegetable retan leathers are more effectively treated after aging. Apparently the chrome is stabilized with time, and possibly some of the sulfated emulsifiers are hydrolyzed.

CHARACTERISTICS OF TREATED LEATHERS

The sterol and the sulfonate compositions produce leathers with physical characteristics which are similar to those of the conventional stuffed sides used for shoe uppers, with the exception that they are a little softer and more flexible. Tests of water vapor permeability (5) show that the breathing qualities of the former are slightly better. The flesh sides appear to have an affinity for water. This permits the absorption of perspiration and contributes to the comfort quality.

The treatments are effective over a wide temperature range, 32° to 100° F., and they appear to remain effective over a broad period of time. The sulfonate treatment appears to be superior in this respect. Six pieces of Army retanned, drum-stuffed leather tested with the Hopton Tester in 1951 showed an average of 4850 flexes, and on retesting in 1958 an average of 5650 flexes before penetration.

The effectiveness of the treatment varies over the area of a side or skin. The results are usually lowest along the backbone, highest in the center portions of the bend and the shoulder areas, and fairly good in the belly area. In the head and in the flanks the results are variable. Water resistance varies from skin to skin within treated lots. Exact causes of these variations are unknown but are attributed to differences in hides and skins themselves

Pieces of sulfonate-treated leather may run as high as 100,000 flexes before penetration on the ALCA (Maeser) machine. A few sides from the same lot may, however, give low results, and some specimens may test as low as 2000 flexes. The average, of course, is moderately high and represents a substantially improved water resistance over fatliquored or conventional stuffed sides.

The treated leathers when tested dynamically show good moisture and chemical resistance against soil slurries, and calcium chloride, salt, and acid solutions but poor resistance against soda alkali solutions.

TESTS

The Hopton Tester (7) was used to determine the water resistance of the treated leathers. A view of an 8-unit tester is shown in Fig. 1.

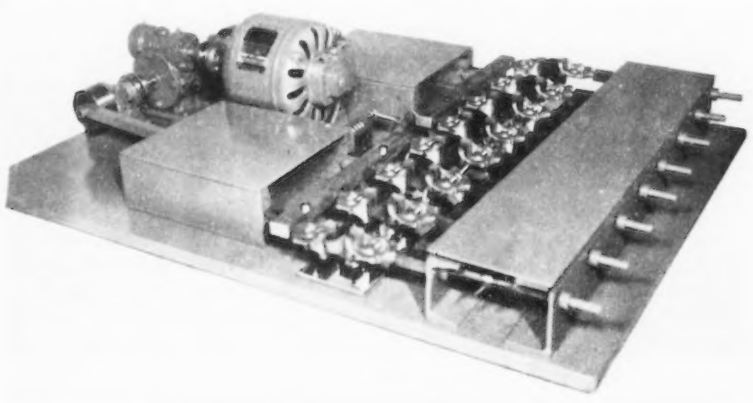

FIGURE 1.—A view of the Hopton Tester.

The specimen is $2\frac{7}{8}$" square and is inserted in the clamp, grain in, with backbone direction perpendicular to the direction of flex. Horizontally placed clamps hold and compress the specimen into the form of a cup, inside which water is held. Pieces of soft neoprene inserted inside the part of the specimen in the clamp make a watertight seal. The drive clamp reciprocates at 120 cycles per minute in a horizontal direction from a distance of 50 mm. to 20 mm. from the stationary clamp. The latter clamp is adjustable and spring-controlled, and limits the force to approximately $7\frac{1}{2}$ pounds on the specimen and compensates partially for differences due to thickness and flexibility. The cup-flex folds are described further in a method for measuring flexibility (6).

The Hopton Tester is more severe than the ALCA (Maeser) tester and penetration occurs in approximately one-fourth the number of flexes. This is due in part to sharper folds, and in part to the fact that water is placed inside the cup rather than outside. In some water penetration tests made several years ago, the author was surprised to find that penetration takes about twice as long when the water is outside the cup, rather than inside.

All specimens were cut from the middle-of-the-bend area and treated directly by application of solvent solutions. The leathers were unselected commercial chrome leathers, and details of tannage and fatliquor were unknown. Penetration readings were taken for each side of the cupped specimens, and the results given in Fig. 2 and Table I are averages for two pieces of each type and amount of treatment.

After the pieces were tested, they were allowed to dry out. After two weeks they were retested in the same way, and this procedure was then repeated a second time. Average results for these tests are shown in Table I together with the percentages of the compositions added to the specimens and data on pore space.

The pore space was determined by conventional liquid saturation methods on degreased specimens. Deodorized kerosene gave the same results as a polar liquid consisting of oleic acid and *o*-dichlorbenzene, and the results were assumed to correspond to the total space readily available to stuffing and treating materials.

In calculating the pore space filled as shown in Table I, an average figure of 80 cc. per 100 g. of degreased leather was used, and an average of 7% was used as the original oil and grease content.

Water vapor permeability tests were made by ALCA Method E32. The compositions described in this paper were compared at different levels in various leathers with conventional stuffed leathers.

RESULTS AND DISCUSSION

The water resistance of treated and untreated specimens from 11 different leathers is compared in Fig. 2. The sulfonate-treated leathers (D) give higher

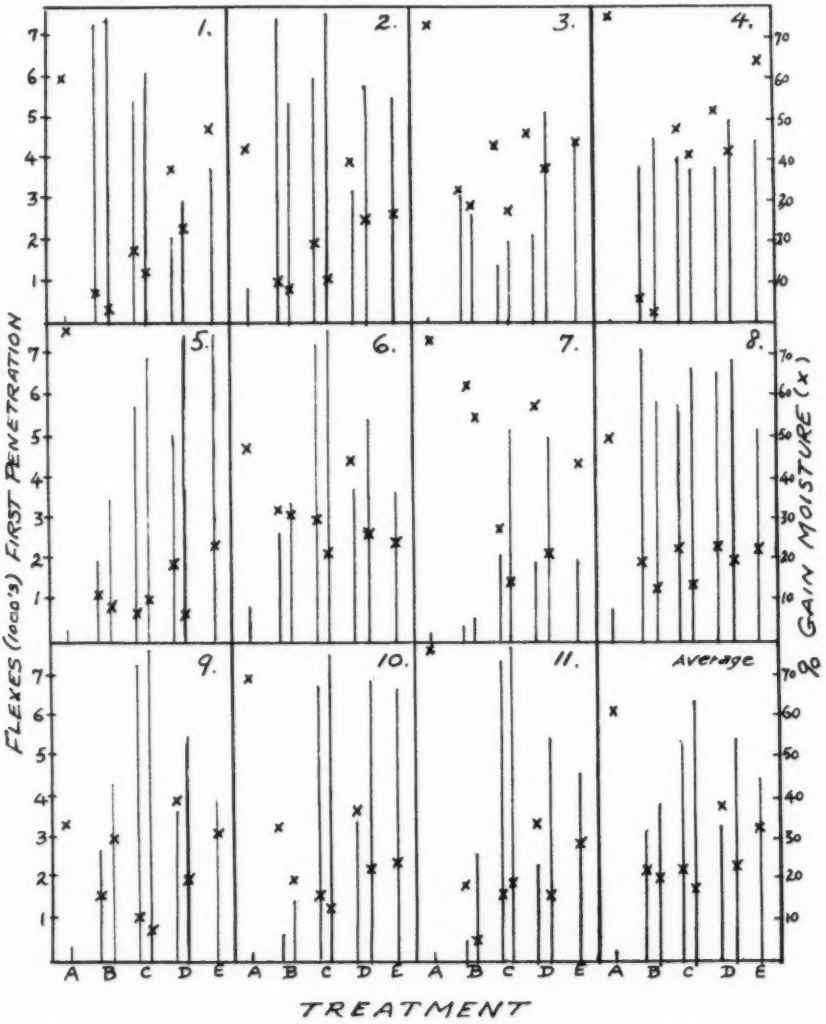

FIGURE 2.—Flexes to penetration and water absorption of different leathers with different treatments tested with the Hopton Tester. (Moisture absorption was determined at 7500 flexes. Results are averages for both sides of two specimens.)

LEATHERS (Chrome shoe upper)—1: calf; 2, 3, 6–9: heavy regular commercial side leathers; 4, 5, 10, 11: heavy medium commercial side leathers.

TREATMENTS—A: no treatment; B: Dow Corning Silicone; C: Bayon 66 (B and C treatments, first vertical line 9% in leather, second line 12% in leather); D: sulfonate composition (first line 9% in leather, second line 15% in leather); E: sterol composition, 15% in leather. Percentages represent deposition in leather on final finished weight basis.

resistance to penetration and lower moisture absorption than the sterol-treated leathers (E). Both give substantial improvement over the untreated leathers (A) or conventional stuffed side leathers which give similar results.

TABLE I

WATER RESISTANCE OF TREATED LEATHERS

(Averages for 11 leathers)

Treatment	Treating Material in Finished Leather by Wt., %	Pore Space Filled by Oil and Treatment, %	Flexes to Penetration			Moisture Gain at 7500 Flexes, %		
			Original Test	Retest 1	Retest 2	Original Tests	Retest 1	Retest 2
A. No treatment	0	10	319	265	629	60.7	66.0	57.5
B. Dow Corning Silicone	12	28	3750	3396	3066	17.7	15.4	12.9
C. Bavon No. 66	12	28	6191	5523	6111	16.4	12.7	14.4
D. Sulfonate composition	15	35	5548	4035	4087	23.9	25.9	25.5
E. Sterol composition	15	35	4591	2383	3159	33.5	37.4	32.8

Treatments with Dow Corning Silicone (B)* and Bavon 66 (C)* give higher water resistance and can be used in lower amounts than the sulfonate or sterol formulations. Treatments B and C also give a few more instances of no penetration at the limit of 7500 flexes and therefore, if the tests were run to completion, the results for these treatments would average a little higher than those given. Although the silicone treatment usually produced the driest or most water-repellent individual fiber, this characteristic is not always reflected in the penetration results.

Leathers which give low penetration results with one treatment usually do so with other treatments. Thus, leathers generally can be classed as difficult or easy to treat effectively. Leather #2, for example, in Figure 2 is easy to treat effectively, whereas leather #3 is difficult to treat effectively.

In the opinion of the author the water resistance of leather treated with sulfonate or sterol compositions depends on the presence of some interfiber, uncombined or unadsorbed sulfonate or sterol. Various tests on upper leather where only the fibers have been treated by chemicals to increase water repellancy give low results for water penetration. The leather invariably allows passage of water under dynamic conditions due to the pumping action of the pores. The presence of some interfiber material is necessary. When water contacts leather treated with these compositions, a degree of

*It should be remembered that the manufacturers of these products undoubtedly have recommendations as to tannage and methods of application which must be followed to attain the most effective use of their products. The tests reported in this paper should not be taken as any basis for commercial comparison.

absorption of moisture into the interfiber composition occurs under dynamic conditions. Moisture is isolated, and the combination functions as a hydrophobic barrier.

For optimum effectiveness of treatment the total content of oils and sulfonate or sterol compositions in the leather should be in the range of 17–22% by weight. In upper leather the pore space needs to be approximately one-third filled.

The sulfonates and sterols are attracted strongly to the chrome or retanned fibers and some combination or entry of sulfonates into the chrome complex is indicated. The combining ability of the sulfonates can be appreciated from the fact that some of the syntans are sulfonates of naphthalene or other hydrocarbons of low molecular weight.

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Received March 15, 1958.

NEW SPRAY SYSTEMIC CHEMICALS TO HELP IN BATTLE AGAINST GRUBS

Announcement has been made that Co-Ral (Bayer 21-199), a spray of systemic chemicals, has received approval, under the food and drug laws, to be used in the control of grubs, hornflies, lice, etc. Several orders for the new product have already been filled. It is expected that this will be a very effective method of grub-eradication.

Co-Ral, a 25% wettable powder, is registered by the U. S. Department of Agriculture for use on beef cattle, horses, sheep, goats and swine. Cattle, horses and hogs are to be treated with a spray dilution of 16 pounds of Co-Ral 25% wettable powder per 100 gallons of water; while 8 pounds to 100 gallons is the dilution for use on sheep and goats. Co-Ral is used only as a spray—not as a dip or oral drench. It is not yet registered for the treatment of dairy animals.

For the control of lice, ticks, screwworms and hornflies, Co-Ral is applied to wet the animal thoroughly without run-off, requiring one quart to one gallon per head as determined by size and thickness of the hair coat. For control of the larval stages of cattle grubs, the spray is applied for thorough coverage and maximum wetting—an average dosage of one gallon per head. Maximum effectiveness against grubs is obtained from single treatments when cattle are sprayed soon after all heel fly activity has ceased—and preferably three months before the first grub holes appear in the back. Obviously, Co-Ral treatments made against hornflies, screwworms, ticks and lice on cattle are effective for cattle grub control, depending upon the time of application in relation to the grub life cycle.
—NHA Bulletin, May 22, 1958.

HAIR ROOT STUDIES*

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ABSTRACT

Both in keratolytic and enzymatic unhairing, hair loosening commences with an attack on the outer root sheath and is continued by the swelling and breakdown of the inner root sheath and parts of the hair that are not keratinized. Papillary hairs, club hairs, and new hairs are affected similarly. Short hairs that project only slightly from the epidermis are not efficiently removed by the unhairing machine. Inefficient removal of the remains of hair roots during scudding leads to leather defects when the hair roots or new hairs are pigmented.

INTRODUCTION

This paper presents some observations that were made in the course of a study of technical dehairing. The question that we set out to answer ran as follows: What effect does the difference in fastness between papillary hairs and club hairs have on technical unhairing? The club hairs ought to be easier to remove than the papillary hairs, because the latter, with their thick, bulblike roots, are anchored much more deeply in the skin.

In speaking of technical unhairing, we refer, naturally, only to the various "hair-saving" processes of the tanner, not to the procedures of complete hair destruction in which alkaline sodium sulfide is applied to the skin in such high concentration that the hair is transformed completely into a gelatinous mass in a short time.

The hair-preserving procedure employed by the tanner consists either of application of very dilute solutions of keratolytic-active materials—sodium sulfide is almost always used for this purpose—or else allowing the same materials, in much higher concentration, to diffuse into the skins from the flesh side only. Thereby only the hair roots are destroyed, without damaging the hair shaft whose conservation is all that matters. This latter procedure is known as painting.

*Republished by permission of the author from *Das Leder*, **8**, 211–22 (1957). Translated by H. B. Merrill. We gratefully acknowledge the generosity of the publishers of *Das Leder* in lending us the cuts from which Figures 2 through 19 were printed.

However, the tanner has at his disposal another type of hair-saving depilatory agent—namely, certain proteolytic enzymes—which far surpasses, with respect to conservation of hair, the keratolytic-active sulfides and analogous active sulfur compounds of the thioglycolic acid type. This type of hair-loosening enzyme has now proved itself to be a valuable assistant in hair root studies. With the aid of such enzymes it is possible to loosen the hair in such a state of conservation that not only are the roots preserved intact but also the glands pertaining to the hair—that is, the fat glands and the discharge ducts of the sweat glands—can be pulled out of the skin along with the hair.

This procedure for isolating morphologically intact whole hairs assumes special interest if one wishes to study the changes that hairs undergo, that is, the transformation of papillary hairs into club hairs and the problem of the regeneration of hairs when club hairs fall out. If the removal of the hair is done carefully, at just the right stage of hair loosening, it is possible to extract the new growth emanating from the clublike roots—the beginning of the replacement hair—along with the club hair. By the use of this rather primitive preparation technique, which however yields surprisingly complete and clear pictures, one can find a new approach to the much-studied, but only partially solved, problem of hair metamorphosis. We have barely touched the outskirts of this problem, which far transcends our original objectives, not to mention our work possibilities. In our attempts to isolate intact hair roots we incidentally obtained examples of different stages of hair degeneration and regeneration, which prompted an orienting survey of the literature on the process of hair metamorphosis. In this paper we shall show a few characteristic photographs, with comments concerning the hair metamorphosis problem, because, so far as we know, these stages of hair renewal have never been illustrated in the very extensive pertinent literature, primarily the literature of zoology and histology. We ourselves were more interested in studying the behavior towards keratolytic agents of intact hair roots of different ages and different stages of regeneration, isolated by the enzymatic procedure. Here too we shall present only a few examples of the results of these studies, without trying to deal exhaustively with the very complicated behavior of the hair roots toward technical depilatories.

MATERIALS AND METHODS

The following hair root studies were done exclusively on salt-cured cattle and calfskin. The depilatory agent used was the enzyme preparation Arazym (Rohm & Haas) which has been recommended for practical unhairing of various kinds of skins. This powdered material, in amount equal to 4% of the soaked weight of the skin specimen, was sprinkled on the flesh side of the soaked skin, moistened with a few drops of water, slicked out uniformly, and lightly rubbed in. If the skin pieces thus treated are stored in petri

dishes, hair side up, at room temperature, the degree of hair loosening can be determined at intervals by pulling out tufts of hair with pincers. Only a very general statement can be made regarding the most suitable digestion period because the rate of action of the enzyme depends on the temperature, the thickness of the skin, and its state of preservation. This optimum digestion period varies from 4 to 20 hours. The degree of hair loosening for obtaining most complete individual hairs is far below the degree of loosening that is wanted in practical unhairing. If the hairs are still too tightly seated in the skin, the regenerating hair elements will not be pulled out along with the hair, while if the hair loosening goes too far the soft parts of the hair roots will be broken down so extensively that one will obtain only the bare, keratinized portion of the hair, without the non-keratinized parts of the root sheaths.

If an enzymatic depilatory is not available, one can obtain good hair root preparations by subjecting pieces of skin to putrefaction—i.e., autolytic and bacterial fermentation—in water at room temperature for from 4 to 8 days. This procedure is very similar to the technical unhairing of, for example, sheepskins by the so-called sweating process.

To investigate the action of sulfide-containing depilatories on the hair roots, single hairs, as complete as possible, obtained by the enzymatic hair loosening, were examined on microscope slides. A 1% solution of sodium sulfide was applied at the edge of the cover glass and allowed to diffuse through the preparation.

THE DIFFERENT AGE STAGES OF HAIRS

Formation of new hairs.—The current conception of the mechanism of hair evolution as represented in the German literature is shown in the following schematic diagram (Fig. 1). The top row represents embryonic hair development, and the bottom row the metamorphosis of hairs to form club hairs and the formation of replacement hairs.

Embryonic hair development is not difficult to comprehend. A pluglike aggregation of cells of the embryonic epidermis intrudes into the little-differentiated connective tissue of the corium. The lowest cell layer of this epidermal appendage later becomes the "matrix", the mother body of the future hair. This matrix enfolds an aggregation of connective tissue cells from which the hair papilla—that is, the connective tissue nucleus of the hair bulb, traversed by blood vessels and nerves—is formed. The embryonic origin and structure of such a papillary root, with its two root sheaths and the papillary hair that grows up from it, are described in all textbooks and handbooks of histological anatomy and therefore may be assumed to be known (2).

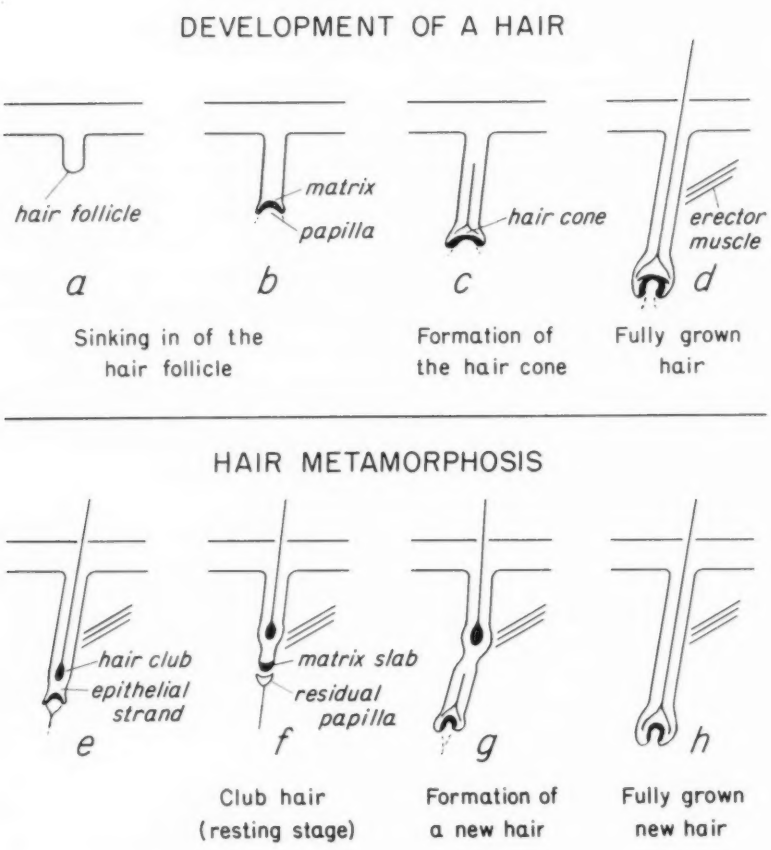


FIGURE 1.—Schematic diagram of hair development and metamorphosis. Taken from Weissenfels (reference 1, page 403), after R. Danneel, *Ergeb. Biol.*, **18**, 55 (1941), slightly modified.

After the growth of the hair stops, the papillary root transforms itself into the club root, whose lower end lies nearer to the surface of the skin than does that of the papillary root. We shall come back to this transformation process later. After a resting period the new formation of a replacement hair proceeds from the club hair, or more exactly stated, from a pluglike, downward-growing aggregation of cells that corresponds to that of the embryonal hair rudiment. This is found beneath the club hair root and is designated in the literature as the "epithelial strand".

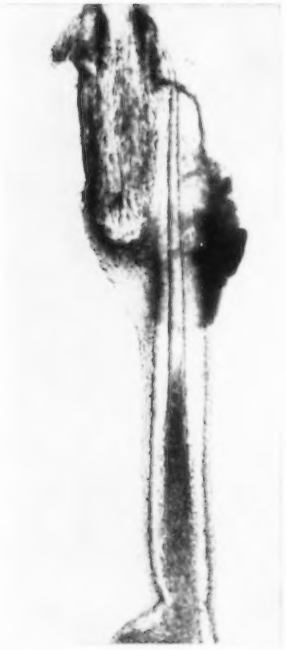
FIGURE 2 (*left*).—Resting club hair with "epithelial strand", fat glands, and sweat gland duct. (approx. × 90)

FIGURE 3 (*right*).—Incipient formation of replacement hair. Notice the broadening of the outer root sheath of the old hair. (approx. × 90)

Figure 2 shows a photomicrograph of a club hair root, which was extracted undamaged from the hair follicle by means of enzyme action, along with its dependent "epithelial strand". A hair root thus constituted is in the resting stage and can be looked upon as the permanent form of the hair. The formation of the replacement hair, proceeding from the epithelial strand, takes place in the same way as the formation of an embryonic hair.

Figure 3 shows a stage of the replacement hair formation in which the new hair tissue has increased in size but is still undifferentiated. Only the lower end is already transformed to the capsule-shaped form of the young hair bulb. Especially noteworthy in this picture is the one-sided widening of the outer root sheath of the club root of the old hair. In this stage it is already evident that the replacement hair will be pointed alongside of the club end of the old hair.

A still later stage, recorded in Fig. 4, shows the already completely developed young hair, whose tip reaches to the height of the fat gland orifice of the old hair. Only a little older is the stage illustrated in Fig. 5. Here also



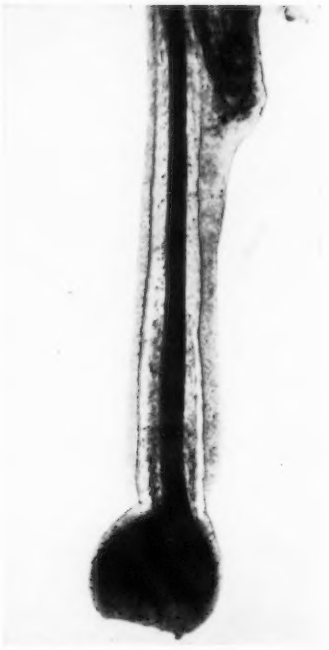


FIGURE 4 (*left*).—A later stage of a replacement hair. Notice especially the common epithelial root sheath of the club hair and the replacement hair. (approx. × 90)

FIGURE 5 (*right*).—A later stage of a replacement hair. The organization of the inner root sheath of the replacement hair into Huxley and Henley layers is clearly recognizable. (approx. × 90)

the young hair, although further developed than in Fig. 4, is combined as a unit with the old hair and its club root through a common epithelial root sheath.

In these young replacement hairs the structural elements of papillary hairs can be recognized especially clearly. Thus in Fig. 5 the differentiation of the cornified inner root sheath into the inner Huxley and the outer Henley layers is noteworthy, a differentiation that in the later stages of development of papillary hairs can be noted only in the immediate vicinity of the hair papilla.

Figure 6 gives an enlarged cross section of a young, unpigmented papillary hair taken close below the orifices of the fat glands where the Huxley and Henley layers become similar and closely fused together. Especially noteworthy here is the so-called "sheath cuticle", that of the innermost root

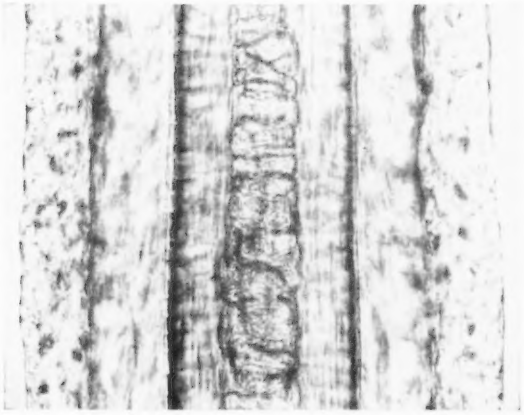


FIGURE 6.—Section of a replacement hair in the neighborhood of fat gland orifices. Optical cross section. (× 355)

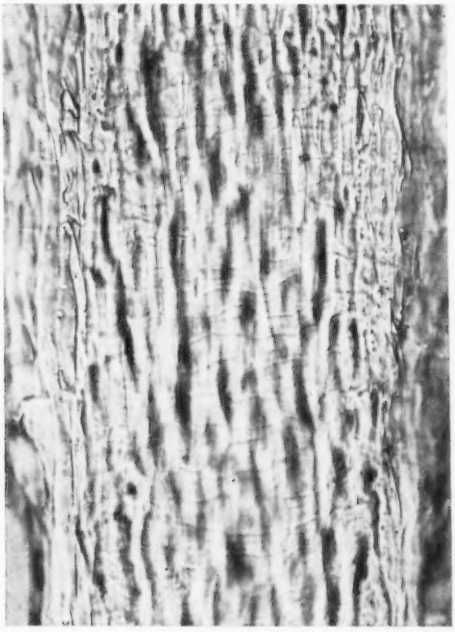


FIGURE 7.—The root sheath of a papillary hair spread out flat in transmitted light. Notice the surface markings of the sheath cuticle as well as the transparent spindle-shaped cells of the Huxley and Henley layers. (× 380)

FIGURE 8 (*left*).—A club hair with completely intact outer root sheath which widens out funnel-fashion above the fat gland orifices. (× 80)

FIGURE 9 (*right*).—Transition stage between papillary and club hair. The hair bulb is missing. The keratinized inner root sheath now surrounds only the upper half of the hair root. (× 85)

sheath layer lying against the "hair cuticle", which is especially clearly recognizable on the left side of the figure. We see that the flat cells of the sheath cuticle that overlap each other like roof shingles interlock with the similarly constructed surface of the hair and in this manner provide the growing papillary hair with an especially firm anchorage in the skin. Obviously any tensile stress exerted on the growing hair will be transmitted to the stable, keratinized inner root sheath, in order thus to protect the growing tissue of the hair bulb.

This scaly arrangement of the sheath cuticle is shown still more clearly in Fig. 7. This shows a part of the root sheath that has been separated from a papillary root and then spread out flat. In this picture we are looking at the inner surface of the sheath whose surface marks are the imprint of the surface of the hair.

The stages shown in Figs. 4 and 5 are encountered frequently in calfskins with the help of the preparative methods already described. Invariably we find that the replacement hair is located in a common outer root sheath

alongside the old hair. It can be seen that the new hair can emerge from the follicle while the old hair still remains anchored in its so-called "club bed". Our plucked specimens afford no support for the statement often found in the literature that the replacement hair pushes the old hair out of the follicle.

Figure 8 is a picture of a club hair root which we succeeded in isolating along with the funnel-like widening of the outer root sheath at the upper end of the follicle. In this case, however, the other appendages of the club root—fat glands, sweat gland duct, and epithelial strand—have remained behind in the tissue. Figure 8 is interesting in that it reveals the dying out of the hair growth in the last stage of activity of the papillary root. The last-formed, lower part of the club hair contains no medulla and is notably thinner than the part of the hair further up the shaft, which belongs to a previous growth stage.

The change of papillary hairs into club hairs. —The process of hair regeneration, proceeding from the epithelial strand of the club root of the old hair, is designated in the American literature as "anagenesis", as opposed to "katagenesis", that is, the changeover of the active hairs to the resting stage of the club hairs (3). It seems that katagenesis is completed very rapidly, at any rate much more rapidly than anagenesis, because when we pluck single hairs we are much less likely to encounter the stage of Fig. 9, which apparently is to be ascribed to katagenesis.

Although a great deal of work has been done on the evolution of papillary hairs into club hairs, much obscurity still rests over this stage of hair regeneration. R. Danneel and his school (4) represent the viewpoint that after the cessation of cell proliferation in the matrix, the hair substance, consisting of a mass of protoplasm composed of still partially non-keratinized cells, is pushed upwards while being gradually keratinized until its lower end is at the level of the point of attachment of the hair muscle. The mechanism of this extrusion of the hair is explained as being due essentially to the pressure of the surrounding tissues, to which the hair root no longer opposes a counter-pressure because of the cessation of cell reproduction in the matrix. Besides this, the matrix secretes a few cells that are no longer differentiated as hair cells but, on the contrary, push the hair upward, whereby the lower end of the mass of hair substance is lifted off the matrix. According to this theory of katagenesis the matrix, which remains unchanged during the rebuilding of the hair root, is pulled upward along with the hair root and forms the outer layer of the epithelial strand of the club hair. Also, the connective tissue papilla moves up along with the matrix. The pigment-producing cells (melanoblasts) are retained in this papilla during the katagenesis and the succeeding resting stage in order to be available in the matrix, the site of their activity, when the new hair germinates. According to this concept a part of the matrix cells, namely, the matrix tip, is carried over from each hair generation to the next one.

The viewpoint of W. Montagna (3) stands in opposition to the above-described theory. That author represents the conception that the lower part of the papillary hair root—by which is meant essentially the non-keratinized part of the growing hair—degenerates in an "orderly, progressive" manner. According to Montagna the hair regeneration starts from cells of the outer root sheath that are to be found in the upper half of the hair follicle. The matrix is formed anew for each new hair generation.

It is not our intention to go more exhaustively into these complicated relationships of katagenesis, concerning which it is obvious that no generally accepted ideas have developed. However, we think it not unlikely that our method of extracting "complete" hairs loosened by a suitable procedure may contribute to the clarification of this phase of the hair regeneration process.

THE BEHAVIOR OF HAIR ROOTS UNDER THE ACTION OF KERATOLYTIC DEPILATORIES

The degree of destruction of hair roots when a mild, "hair-saving" depilatory acts on skin.—For our purposes the most interesting method of hair removal is that accomplished by a mild "hair-saving" process in which the hides are kept for a considerable time in a lime suspension, low

FIGURE 10.—A clump of hairs extracted from a mildly limed skin. Papillary and club hairs roots are to be found in both the coarse and the fine hairs. (× 85)

in sulfide, with the expectation that the keratolytic action will be limited to the hair roots. A cowhide limed for several days by a hair-saving method gives up its hairs without difficulty when the attempt is made to pull them out. Figure 10 shows the root portions of a carefully extracted clump of hairs which were loosened with sulfide but are still well preserved. One can obtain enough of the roots to make it possible to distinguish between the different age forms—that is, between papillary and club hairs—only if the tension is applied in the direction of growth. If the hairs are removed with a scraper or by means of an unhairing machine having a similar action, the ends of the roots will break off and remain in the skin. It is difficult, if not impossible, to differentiate between the two hair types in hair thus removed.

We see in Fig. 10 that cowhide possesses two kinds of hairs which differ in thickness. The finer hairs, which are less deeply implanted in the skin than the thicker hairs, are called wool hairs. Our attention will, however, be confined to the stronger, normal hairs that make up the greater part of the animal's coat, because the behavior of the roots can be studied more readily with this type.

Examination of this plucked cluster of hairs shows unequivocally that it includes both papillary and club hairs. The papillary root no longer possesses the characteristic hair bulb, which obviously was destroyed or broken off and left behind when the hair was extracted. We can recognize this type of hair root, however, by the characteristic, highly light-refracting inner root sheath that frames the lower end of the hair like a bright boundary stripe. The boundary of the inner root sheath is especially clearly recognizable when dealing with colored, i.e., pigmented, hairs, because the inner root sheath is always free from pigment.

Although in this method of unhairing, the inner root sheath remains intact, nevertheless it is unmistakable that a certain amount of disintegration has occurred under the influence of the depilatory chemicals—that is, a certain degree of separation has taken place of individual, elongated units of root sheath cells, split off from the general aggregate. We may contrast this with the appearance of the inner root sheath of an enzymatically loosened papillary hair root in which no such appearance of dissolution is to be seen if the fermentative action is interrupted at the right time (Fig. 17a).

In the center of Fig. 10 there is to be seen a club hair in its often described typical standard state. The end of this hair lacks the inner root sheath, the hair medulla, and pigmentation, and furthermore the lower end is rounded off club-fashion (*see also* Fig. 2), while the ends of the papillary hairs look as if they had been broken off.

So far as concerns the outer root sheath, hardly a trace of it is extracted from the skin with either type of hair root, and neither is any vestige of the epithelial strand attached to the club root, nor any already sprouted young hair. The absence of these young hair elements from these plucked prepara-

ations should cause us no surprise, because the connection between new hairs and club hairs is provided as was shown previously by the common outer root sheath, whose non-keratinized cell system is especially sensitive to swelling in contact with depilatory systems, as may be seen in Figs. 15b and 16b.

In the case of preparation of club hair roots loosened with a sulfide depilatory we often find a characteristic white, transparent, swollen cellular layer on the surface of the hair at about the level where the medulla ends (Fig. 10, arrow, and Fig. 11). We meet with these cellular structure in limed hairs

FIGURE 11 (*left*).—Segment of a mildly limed club hair at the level of the surface of the skin. (× 340)

FIGURE 12 (*right*).—"Scud" from a mildly limed skin, showing papillary roots, club roots, the contents of fat glands, and spindle-shaped cells of the inner root sheaths. (× 80)

with considerable frequency. Since they are found only on club hair roots and fit the outer surface of the hair closely, the supposition occurs at once that these cells belong to the root cuticle of the innermost layer of the otherwise vanished inner root sheath. However, this is only a speculation.

If a skin that has been limed with a hair-saving depilatory is unhaired mechanically, the root ends, as already mentioned, are for the most part

broken off and not scraped off with the hair shafts. Following this, we try to squeeze the remains of the hair roots out of the pores by a special operation, scudding, in which part of the contents of the fat glands is also removed. By microscopic examination of the mixture expelled from the hair follicles the so-called "scud"—consisting of fat gland constituents and more or less keratinized remnants of hair roots, one can easily establish the presence of papillary and club root residues in close proximity to each other. In Fig. 12 the hair bulb of a papillary root is clearly to be seen, identifiable further by being encased in the inner root sheath. The club root located in the middle of the picture is again identified by its characteristic rounded-off lower end and by lack of an inner root sheath. We can also recognize fat gland remains and individual keratinized cells of destroyed inner root sheaths (Fig. 12, arrow), which are never lacking in a smear of scud.

The degree of destruction of the hair roots during the action of a depilatory paint on the hide.—If we extract clumps of loosened hair from painted skins—calfskin, for instance—in the same manner as from mildly limed skins, the differentiation of club hairs and papillary hairs becomes much more difficult. In this case all the hair shafts terminate in a smooth broken surface, so that one criterion of club hairs, the rounded lower end, has disappeared (Fig. 13).

FIGURE 13.—A clump of papillary hairs from a painted skin (early stage of painting) showing root sheaths, epidermal cells, and sweat gland ducts. (× 85)

However, by closer examination of the generally amorphous-looking mass of strongly swollen root sheath residues that lies around the ends of the hairs, we can make certain statements. Those hair ends that came from papillary hairs are surrounded by a clearly organized layer of partly swollen cells that doubtless came from the inner root sheath. Moreover, in the keratinized elements of this root sheath we can still recognize cell outlines, and thus the characteristic elongated spindle shape of these cells. However, it remains for the future to establish with certainty a connection between these still largely organized cells and an inner root sheath.

FIGURE 14.—A papillary hair root from a painted skin. (× 400)

Figure 14 shows the end of a papillary hair, estimated to be broken off in the middle of the root sheath, under higher magnification in order to make the construction of the cell mass of the root sheaths clearly recognizable. In Fig. 13, we can recognize canal-like channels in a few places in the swollen cell mass surrounding the hair end; these probably represent sweat gland discharge ducts (*compare with* Figs. 2 and 3).

Finally, it is very typical of hair that has been loosened by painting that a keratolytic action of sulfide on the hair shaft is already recognizable in the form of a slight broadening by swelling and a slight S-shaped curvature of the lower hair ends.

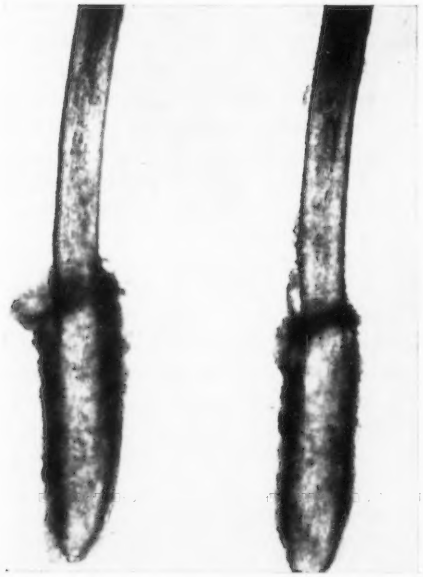


FIGURE 15a.—Club hair roots loosened by enzyme action. (× 80)

FIGURE 15b.—The same club hair roots swollen in 1% sodium sulfide solution. (×80)

Swelling phenomena in isolated club and papillary hairs. *Club hairs.*—To observe better what happens to hair roots when they are acted upon by sulfide, without having to study the depilatory action on hairs that were previously subjected to keratolytic attack, we have subjected hairs that were loosened by enzyme action,with root ends as nearly intact as possible, to the swelling action of dilute sodium sulfide under a microscope slide cover glass, and observed what took place by means of the microscope.

Figure 15a shows two club hair roots as they appear after isolation from the skin. Figure 15b gives the swelling picture of the same hair roots in sodium sulfide solution. The club roots, when removed from the skin by the previously described method, are still enveloped in a layer of cells, the "club bed" which is generally regarded as pertaining to the outer root sheath. However, from Fig. 15b it can be inferred that the elements of this club layer are not of a uniform nature. In the swollen state two layers can be distinguished, of which the inner one is manifestly less swollen than the outer. Perhaps in this inner layer we have a residual constituent of the inner root sheath.

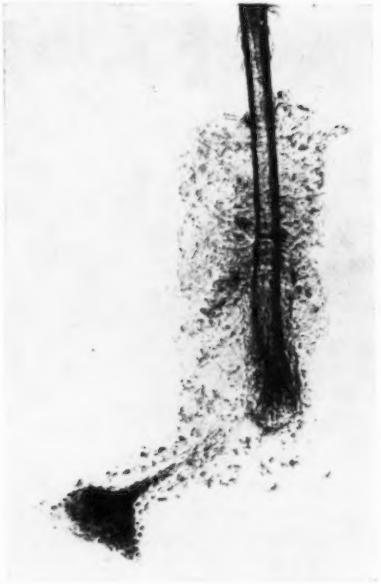


FIGURE 16a (*left*).—A club hair, with replacement hair attached, loosened by enzyme action. (× 100)

FIGURE 16b (*right*).—The hair regeneration stage of Fig. 16a swollen in 1% sodium sulfide solution. (× 100)

That the root sheath of the club hair root is not uniform throughout has been emphasized also by Montagna (3, p. 179), in that he describes a partially keratinized "capsule", one or two cell layers thick, between the epithelial root sheath and the club root, which capsule he, too, considers to pertain to the inner root sheath.

When one subjects a club hair with a dependent replacement hair (Fig. 16a) to such a treatment with dilute sodium sulfide solution, it becomes evident, as shown in Fig. 16b, why even a very mild keratolytic action makes it impossible to extract the replacement hair from the skin along with the old hair. The sensitive cell mass of the outer root sheath, which binds the old and young hairs together as a uniform, undifferentiated cell system, immediately undergoes an enormously strong swelling, so that the connection between the two hairs is dissolved.

No matter how carefully the thus loosened hair is extracted—not to mention technical unhairing—the new hair will remain in the skin. If the young hair is a carrier of hair pigment, as is the case with those in Figs. 16a and 16b (the associated old hair is, on the contrary, pigment-free because on cessation of growth of the papillary hairs the production of pigment through the melanoblasts of the matrix comes to an end earlier than the formation of hair cells), then damaging, because pigment-containing, hair root residues will remain behind in the skin, residues which are very difficult to remove because they are so deeply embedded in the skin, and because they are disconnected from the mechanically removable old hair shafts.

Papillary hairs.—If isolated papillary hairs are studied in the same way (Figs. 17a and 17b), we observe two interesting swelling phenomena. The first pertains to the inner root sheath which, as mentioned before on page 455, already responds to a mild keratolytic influence. When the inner root sheath is freely exposed, this keratolytic action of a dilute sodium sulfide solution brings about an immediate strong swelling of the root sheath without at first destroying the connection between the individual cells. The sheath detaches itself all around the hair shaft and expands, so that a moderately large intermediate space is formed. This interspace is clearly recognizable in Figs. 17b and 18, in that we can recognize alongside the cutting of black-pigmented hair a fragment of the inner surface markings of the sheath cuticle (*see also* Fig. 7). It is self-evident that this swelling of the inner root sheath contributes in an essential manner to the loosening of the papillary hairs when we recall that the tight interlocking between sheath cuticle and hair surface is the most important means whereby the papillary hair is anchored in the skin.

In Fig. 17b we can note further that the lower ends of the papillary hairs, which we know to be in an intermediate stage of keratinization, are likewise subject to swelling and have become thicker. This thickened zone is not

FIGURE 17a.—Papillary hairs loosened enzymatically. (×80)

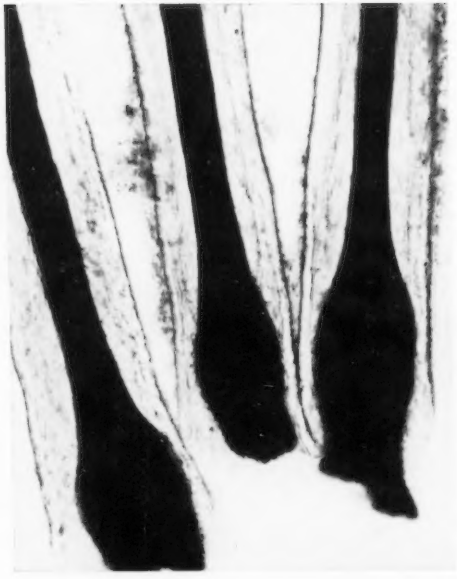


FIGURE 17b.—The papillary hairs of Fig. 17a after swelling in 1% sodium sulfide solution. (× 80)

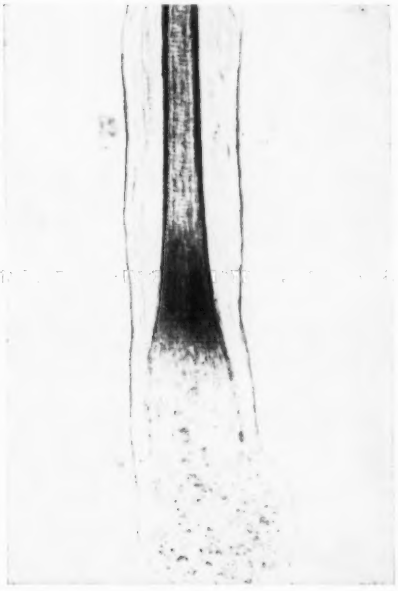


FIGURE 18.—An unpigmented papillary hair, with bulb, swollen in 1% sodium sulfide solution. (× 80)

to be confused with the hair bulb, which would be located below this swelling, and which remained stuck in the skin in the case of the material used for Fig. 17 (*compare in this respect* Fig. 17b *with* Fig. 18). The fact that the thickening brought about by the swelling of this part of the hair is not uniform but reaches a maximum at one particular place and there fills completely all the intermediate space provided by the expanded inner root sheath probably is brought about by the progressively different degree of keratinization.

However, when the papillary roots are in place in the skin (rather than isolated from the skin) the development of such a swelling is possible only to a limited extent. All through this research it has been shown that the papillary root, including the true hair parts as well as the keratinized root sheaths, is highly sensitive to keratolytic action. Under the conditions of practical unhairing with a mild depilatory this results in a relaxation of the tight contact between the structural elements of the hair and the inner root. Simultaneously, the still non-keratinized part of the papillary root is so weakened that the upper part of the hair root breaks off from the hair bulb on slight tension.

FIGURE 19.—The detached inner root sheath of a limed papillary hair. (× 345)

A more prolonged action of weak sodium sulfide solution brings about a dissolution of the bonds uniting the keratinized individual cells of the inner root sheath, so that the inner root sheath, which is already expanded by swelling, breaks up into its component cells on gentle mechanical action (Figs. 10 and 12). Figure 19 again shows clearly, in the case of a limed papillary hair root, how the application of pressure to the cover glass separates the sheath cuticle from the rest of the inner root sheath, while the individual spindle-shaped cells of the Huxley and Henley layers separate from each other.

DISCUSSION OF THE RESULTS

The question that was posed at the outset, whether the more deeply anchored papillary hairs are more difficult to remove in tannery practice than the less deeply rooted club hairs, can be answered in this manner: No difference in ease of removal of these two hair types is to be found either in enzymatic or sulfide unhairing. As soon as the non-keratinized—and therefore highly sensitive to swelling—outer root sheath is attacked by one means or another, the hair is loosened.

However, as keratolytic hair loosening is carried out in tannery practice, other parts of the root—namely, the inner root sheath which is susceptible to swelling in spite of being keratinized and the lower parts of the papillary root itself in various stages of keratinization—are attacked and weakened, so that at least the papillary hairs are broken off at the lower part of the root by mechanical scraping action. Even the club hairs usually break off under a scraping action, leaving the butt end behind.

Thus, remains of roots are left in the skin; these give rise to trouble only if they are pigmented. From the tanner's standpoint there is no objection to this leaving behind of the spindle-shaped cells of the inner root sheath and the lower end of the club root, because these parts are invariably pigment-free. However, the leaving behind of pigmented parts of papillary hair roots and the pigmented young hairs found beneath the club hairs may well cause trouble. Complete removal of these pigmented remains of hairs from the skin has never been successfully accomplished, either by mechanical means (striking out) or by chemical methods (modification of the swelling of the skin).

The un-uniform coloration of natural-colored leathers or those dyed with aniline dyes of light shades—referred to as "irregular" or "cloudy" by technicians—is brought about in part by these residues of pigmented hair roots.

Even in the case of enzymatic unhairing, where at least the keratinized parts of the roots are scarcely attacked, the complete removal of pigmented root constituents along with the hair is scarcely possible in tannery practice. This holds true especially for the replacement hair assembly in which a strong pigment formation in the hair bulb has already set in. The removal of the hair root remains left behind in the skin by enzymatic hair loosening naturally occasions just as much difficulty as in the case of keratolytic hair loosening.

Besides the problem of removing pigmented hair root residues, that of "ground hair" gives the tanner the most trouble. By this we mean young hairs that have grown until their tips are already a few millimeters above the upper surface of the skin and which often are not removed in hair-saving depilation. The tanner usually first notices these ground hairs at a later stage of leather manufacture, when chemical destruction of these hair points is no longer possible for technical reasons. In these circumstances the troublesome hair tips must be shaved off with a razor blade. Since the young hairs in this phase of growth certainly do not react toward unhairing materials any differently than hairs of a somewhat later growth stage, which are easily removable, the cause for the difficult removal of ground hair is merely to be sought in the fact that the very fine hair shafts are not gripped by the scraper blades of the unhairing machine, or not gripped sufficiently. No proof exists of the often expressed supposition that the keratin of the ground

hair in contrast to fully grown hair is "immunized" by a faulty liming procedure, that is, made insensitive to the action of the depilatory chemicals, for according to experience, this sort of ground hair is never observed after a hair-destructive liming.

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2. Küntzel, A. in *Handbuch der Gerbereichemie und Lederfabrikation* (Vienna: Springer-Verlag, 1944) Vol. I, Part I, p. 183.
3. Montagna, W. *The Structure and Function of Skin*. New York: Academic Press, 1956.
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We get glimpses of colonial processes for making leather from the early laws providing that tanners must not overlime, burn, or hot-wooze their hides, and that curriers must not use poor materials, but good hard tallow, "as much as the leather will receive." Even at an early date some colonial tanneries were large establishments for the period and used imported materials. The inventory of a New England tannery, in 1653, showed 415 hides in the bark, valued at about $2000; 45 hides in the lime worth over $100, and 312 West Indian hides in the lime worth over $600. Yet the process of tanning was very slow, and a century later the equipment of even the better establishments was limited to a number of small wooden boxes or vats sunk in the ground near a stream, without outlet or shelter, and either open pools or similar vats above ground for liming. An open shed sufficed for a beam house, while the bark was crushed at the rate of half a cord a day by alternate wooden and stone wheels drawn round a circular trough some 15 feet in diameter. Bark mills are mentioned as early as 1661, and probably were in use previously, though at this period they were seldom if ever driven by water. Some of the first mills seem to have been owned in partnership by several tanners. In New England and New York the principal tanning material was hemlock bark, while the central and southern colonies used oak bark, which made better sole leather. A representative colonial tannery, situated at Trenton, had 64 vats, 5 limes, 2 water pools, a bark house holding 300 cords, a currying shop and a skin dresser's shop, and facilities for making leather breeches. The use of this article for clothing probably explains the introduction of fulling or beating mills, operated by water-power, which were used to render leather pliable and possibly to finish its surface. In 1741 there were "two fulling mills for the fulling of leather" in Dorchester, and among the early water-rights granted at Salem was one for a "wash leather mill." Though colonial tanners were importers of skins and exporters of leather, and their trade extended not only to other colonies but also to Spanish America, we have no record of large companies or of British capital engaged in this industry. The business was not fostered by special privileges, and most of the laws affecting it were restrictive.

— *History of Manufactures in the United States*, Vol. I, 1607–1860. Victor S. Clark, New York, McGraw-Hill Book Company, Inc., 1929.

LIFE LINES

Brief Biographies of Our Contributors

ALFRED W. HOPTON will be remembered as the developer of the flexibility gauge discussed in a paper presented at the Fifty-second Annual Meeting and published in March, 1957. His biography is given in the *Journal* of that date.

DR. ADOLF KÜNTZEL—See the May, 1958, *Journal* for biographical data.

HIDE SHRINK

From time to time we receive questions concerning hide shrink and pack building. Here is what K. E. Nielsen of International Packers Ltd., Chicago, Ill., has to say about them in his splendid paper, "Meat Packing Plant Refrigeration," which deals largely with Hide Curing:

Hide shrink may vary between 12 and 18 per cent. Normal expectancy is as follows:

Native Bull	16-18%
Native Steer	13-18%
Light Cow	14-17%
Calf & Kip	12-15%

The weight of a hide from an animal will vary throughout the year. For example, the hair on the hide is usually short during the late summer and fall, then grows longer during the winter. For a yearly average, it will be satisfactory to assume the green hide is approximately 6½ to 7½ per cent of the live animal weight. Hides from fattened steers will be in the lower bracket, while the hide will be of higher percentage with lean cattle.
—*N. H. A. Bulletin*, January 13, 1958.

FOOT-AND-MOUTH DISEASE VIRUS PHOTOGRAPHED

Scientists at the Plum Island Animal Disease Laboratory of the U. S. Department of Agriculture report that they have purified, isolated, and photographed the foot-and-mouth disease virus.

Their first electron micrographs of the virus reveal that it is spherical in shape and about one-millionth of an inch in diameter. This is the smallest of any of the viruses affecting animals—even smaller than polio virus.

These new findings about the physical make-up of foot-and-mouth disease virus are expected to speed research and development of new and effective means of combatting the disease.

This accomplishment was reported recently by Dr. H. L. Bachrach and S. S. Breese-Jr., of USDA's Agricultural Research Service, at the fall meeting of the Electron Micro, scope Society of America in Cambridge, Mass.
—*Aminco Laboratory News*, Vol. 14, No. 6, November, 1957.

ALCA NEWS

Proceedings, Reports, Notices, and News

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Notice to Authors

Authors are requested to introduce each paper submitted to the *Journal* by an abstract of the article. Articles should also be accompanied by a brief biography of the author(s) for inclusion in the "Life Lines" feature in the *Journal*. Unnecessary correspondence will also be avoided if a notation is attached to the copy indicating where the author desires galley proofs to be sent.

ALCA GROUP VISITS LTI LEATHER LABS

Dr. Albert E. Chouinard, extreme left, demonstrates a shaving machine in one of the laboratories of the Leather Engineering Department which he heads at Lowell Technological Institute to ALCA members who visited LTI during the Fifty-fourth Annual Meeting.

COMMITTEE ON PREPARATORY AND POSTTANNING PROCESSES

Report of Meeting May 25, 1958

The Committee met at Swampscott, Massachusetts, on May 25, 1958.

The G series methods which are under the jurisdiction of the committee were printed over two years ago last September. It is recommended that the G series be accepted as official. This recommendation is made on the basis that, prior to rewriting them, most were official methods accepted by the membership and no objections about the rewritten methods have been made during the period of over two years that they have been available to the members.

Method J40 (Sampling of Tannery Chemicals) will have been in print for a period of two years in November, 1958. Action on this method will be taken at that time.

Method J50 (Sampling of Fats and Oils and Their Products) has been in print since March, 1954. As of May 10, 1956, R. M. Koppenhoefer, former chairman of the Subcommittee on Fats and Oils, recommended that this method remain provisional because it was adopted in part from a method carried in tentative status by a sister technical society. At the present meeting it was decided to make the method official.

W. T. Roddy, *Chairman*

PHYSICAL AND MECHANICAL PROPERTIES COMMITTEE

Minutes of Meeting May 25, 1958

The Committee met at Swampscott, Massachusetts, May 25, 1958, with these members present: Chairman E. D. Compton, R. G. Ashcraft, M. M. Baldwin, R. J. Kanagy, A. N. Kay, C. W. Mann, R. L. Young, and M. Maeser.

The Committee had no specific business since no methods have been proposed by the Joint ALCA-ASTM Committee on Leather. The various members of the Committee reported on their activities as subcommittee or section chairmen of the Joint ALCA-ASTM Committee. Some specific areas of the work were discussed as noted below.

R. G. Ashcraft, Subcommittee III, Section 4 (Measurement of Water Resistance).—This Section is working on resistance of upper leather to dynamic water penetration, resistance of garment leather to water spray, dynamic water absorption of light leathers, resistance of glove leathers to water penetration, water penetration of heavy leathers, static water absorption, and dimensional stability as influenced by water. For resistance of upper leather to dynamic water penetration the Committee is considering both the Maeser tester and the Dow Corning tester.

Mr. Ashcraft and his Section have considered the various water absorption and resistance tests of the International Union. Progress with these methods will be observed, but relatively little work will be done by the Section at this time.

M. M. Baldwin, Subcommittee IV (Research).—This Subcommittee is making a study of abrasion methods and would appreciate any suggestions or help in the two major areas being investigated, namely, non- or sub-destructive tests (including cycling procedures) and test methods for evaluating properties related to comfort.

A. N. Compton, Subcommittee III, Section 7 (Mechanical Leathers).—Mr. Compton reported by letter as follows: "The corrosion test procedure has been approved by letter ballot of the Section members. It has been forwarded to the Secretary of the ALCA-ASTM Joint Committee for letter ballot of the entire Committee and possible subsequent inclusion in the annual report to the ASTM. This test procedure is intended to evaluate chrome leather washers that will be used in hydraulic systems where all sources of possible corrosion must be kept to a minimum, such as in aircraft. Its basis is Specification MIL-R-5521A.

"The packing permeability test is presently in the hands of Dr. Lollar for statistical assistance in determining the degree of precision, following a round robin -type test on 64 packings."

E. D. Compton, Subcommittee III, Section 5 (Lightfastness of Leather).—All of the leathers have now been received, and the sample kits should soon be sent to Dr. Stubbings for the first determination of color values.

A. N. Kay, Subcommittee III, Section 10 (Miscellaneous Physical Properties).—The following methods are under investigation: E40 (Pipe), E43 (Stiffness), and E45 (Compressibility). The method on break will be considered after the work of Mr. Bailey, in Federal Specification KK-L-311a, has been developed further. Some difficulty is being experienced in laboratory-to-laboratory checks on E45. The procedure for evaluation of run of glove leather is giving some difficulty. C. W. Mann requested any suggestions that Committee members might have.

C. W. Mann, Subcommittee II (Sampling and Conditions for Physical Testing).—The method for conditioning of leather has been submitted for publication. The procedure for sampling of side leather is being written. Work is being done on sampling methods for small skins.

Mr. Mann reported that his Committee has reviewed the methods of the International Union on sampling and conditioning and found them little different from the ALCA methods except for the use of 65% relative humidity.

J. Naghski, Subcommittee III, Section 2 (Physical Dimensions).—Dr. Naghski reported by letter as follows:

"This Subcommittee has been working on the following four ALCA methods: E2 (Determination of Area), E3 (Thickness of Units), E4 (Thickness of Specimens), and E5 (Determination of Width.)

"The adoption by the Advisory Committee of a requirement for sections on significance and on precision has made it necessary to revise the proposed drafts of the methods to include these sections. Sections on significance and on precision are being prepared, and the methods will be resubmitted to the Joint Committee as soon as these are completed."

Dr. Naghski reviewed the proposed method for thickness of the International Union in the light of the ALCA method and of the proposed ALCA-ASTM method, and the points of variance were brought to the attention of Mr. Maeser for submission to Dr. Mitton.

In discussion of measurement of thickness it was agreed that two methods for measuring thickness were required, one for heavy and one for light leathers. With heavy leather some difficulty is encountered with specimens that have a curvature in the area where thickness is determined.

R. Stubbings, Subcommittee III, Section 5 (Surface Characteristics). E. D. Compton reported for Dr. Stubbings who was in another meeting. The following methods are under investigation: Cold Test for Upholstery Leather; Flex Test of Leather Finish in Upholstery Leather; Crock Test; Flex Test of Finish in Upper Leather; Lightfastness Test of All Leathers; Scuff Resistance of Leather; Battelle Adhesion Test; Oil Resistance Test; and Stain Resistance Test.

R. Young, Subcommittee III, Section 3 (Tensile Properties). The following tests are under investigation: ball burst, tensile, elongation, tongue tear, and stitch tear (double-hole). The tear resistance test of the International Union will be studied. In general, the above methods are being studied in order to meet the requirements of the significance and precision statements required by Dr. Lollar's Nomenclature, Definitions, and Applicability Subcommittee of the Joint ALCA-ASTM Committee on Leather.

Mr. Mann pointed out that the wire used in the stitch tear test should be more carefully specified than in the ALCA method.

The Committee discussed the ball burst test in some detail. It was noted that for heavy leather a 1/4-inch plunger through a 3/4-inch hole is more a puncture test than a burst test. It was agreed that the test procedure had value but that such a test used on heavy leather should have another title.

After discussion of the relative merits of the Mullen test and the ball burst test, it was agreed that Mr. Young's section of the Joint ALCA-ASTM Committee should consider inclusion of both the Mullen and the ball burst tests, since these tests evaluate different properties of the leather. Also, it was noted that the Mullen test is more widely used in the industry than is the ball burst test.

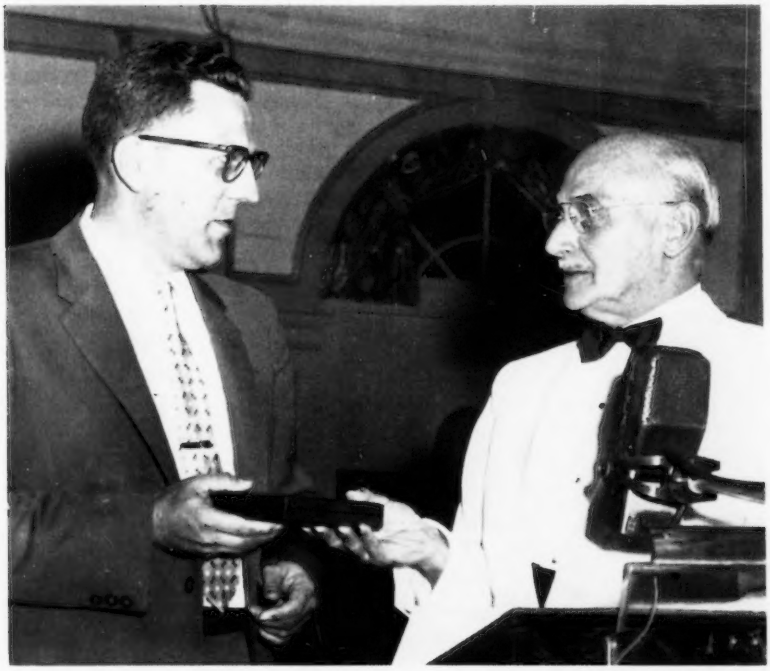
E. D. Compton, *Chairman*

REPORT OF THE SUBCOMMITTEE ON FATS AND OILS

The subcommittee met at Swampscott, Massachusetts, on May 25, 1958. Present were C. A. Blair, B. A. Grota, C. M. Morrison, and C. E. Retzsch. Results on analyses of chrome-retan upper leather for free and combined

oil were discussed. This included a comparison of individual analyses and comparison of results obtained by the previously adopted method, the chloroform extraction method, and the ethyl ether extraction method. Application of the method to other types of leathers was also considered. It was decided that some of the work should be repeated, and when complete, the best method for free and combined oil in chrome-tanned leather will be submitted as a provisional method.

MERRILL LEACH, *Chairman*



Courtesy of The Leather Manufacturer

Dr. Robert Stubbings *(left)* is presented Alsop Award by B. A. Schiller at Fifty-fourth Annual Meeting.

ANALYSIS OF VEGETABLE-TANNED LEATHER
CORRECTION TO METHOD B10—GLUCOSE

Par. 5.1.—The factor 200 used for calculating the glucose content is in error when the dextrose present is determined in milligrams, as is usual and called for in the formula and table. This factor should be changed to read 0.2 (200/1000).

ARTHUR N. KAY, *Chairman*,
Vegetable Leathers Committee

PRIZE PAPER FOR SIXTH I.U.L.C.S. CONFERENCE

The Council has voted to award a prize of $1000 to assist in defraying the travelling expenses of the author of a paper to be presented at the Sixth Biennial Conference of the International Union of Leather Chemists Societies, at Munich, Germany, in September 1959. The conditions of the award are as follows:

1. Any active member of ALCA may enter the contest by submitting an original paper not previously presented or submitted for publication elsewhere. The subject of the paper may be any phase of the science or technology of leather manufacture.

2. The award-winning paper will be selected by a committee appointed by the President from the members of the Council and the Editorial Board of the *Journal*. The committee is authorized to reject all entries if, in their opinion, such action is desirable.

3. All papers to be considered for the award must be received not later than March 1, 1959. Each paper submitted must be complete as to content, including tables and or illustrations, and substantially complete as to form.

4. Manuscripts should be mailed to the Editor-in-Chief of the *Journal*, with a covering letter stating that the manuscript is to be entered in the prize competition. The Editor will turn over all such manuscripts to the chairman of the award committee.

5. Announcement of the award-winning paper will be made not later than May 1, 1959.

6. Papers other than the one selected for the prize will, if acceptable, be published in the *Journal* and may be presented at the 1959 ALCA meeting, if such action is agreeable to the authors and the convention program committee. The prize-winning paper will be published in the *Journal* after it is presented at Munich.

ABSTRACTS

The New Types of Leather and New Shoe Finishes. H. Stock. *Das Leder*, **8**, 289–93 (1957). **Comments from the Viewpoint of the Shoe Manufacturer.** E. Elias. *Ibid.*, 293–94. **Comments from the Viewpoint of the Leather Manufacturer.** F. Wassmuth. *Ibid.*, 294–95. **Discussion.** *Ibid.*, 295–97.—The function of the finishing room is to make salable the shoes that were soiled or damaged during manufacture, and consists of repairing and finishing. Small defects can be repaired with wax preparations. A well-compounded collodion–artificial resin preparation is the safest repair material, even for casein-finished leather. Some shoes with bad two-tone effects cannot be repaired but must be redyed. Before being repaired or finished, the leather must be thoroughly cleaned; then it must be finished at once before dust can settle on it. Finishes were formerly classed as polishes (wax base), dressings (wax and shellac), and finishes (shellac solutions). These terms should be retained even though shellac has been replaced to a considerable degree by cellulose derivatives, artificial resins, and polymers. Mention is made of methods of applying these materials and of new products such as mat polishes and creme polishes. I.D.C.

Production of Chrome-Vegetable Shoulders for Inner Soles. C. Gastellu and I. Jullien. *Bull. Assoc. Franc. Chimistes Inds. Cuir*, **12**, 250 (1957).—Chrome-vegetable-tanned leather is particularly suited for inner soles, since it provides easy channeling, solidity for Goodyear construction, excellent water vapor permeability, and better perspiration resistance. The following procedure is given: *Raw stock:* cattlehide shoulders, 17–20 lb. average green-salted weight. *Soak:* Mill in a drum in 250% water for 1 hr.; wash 1 hr.; refill and soak (still) for 6 hr.; wash again; soak in maximum float overnight. *Lime:* Next day, wash 1/2 hr.; lime in the same drum with 300% water, 12 g. CaO per l., and 3.6 g. Na_2S per l.; turn the drum 5 revolutions every 30 min. from 8 a.m. to noon, and 6 revolutions every hour from 2 to 6 p.m. (drum speed 8 r.p.m.); rest overnight. Next morning, run 30 min.; drain; wash 20 min.; flesh and level; determine white weight. *Deliming:* Wash in drum 30 min.; then run 40 min. in 300% water and 1% NH_4Cl; add 0.5% HCl (Bé. 22°) diluted 1:5; mill 4 to 5 hr. longer; wash 20 min.; drain. *Pickle and tannage:* To the skins in the drum add 70% water and 7% NaCl; run 10 min.; add 1% HCl (Bé. 22°); run 2 to 3 hr.; let stand overnight to pickle completely through. Add Cr liquor equal to 2% Cr_2O_3, 48% basic, in 3 feeds during 3 hr.; mill for 8 to 9 hr. in all; let stand overnight; run 30 min.; make boil test. *Washing and neutralizing:* The importance of this operation is stressed, as it is essential that salt and acid from the tannage be eliminated. Wash 1 hr.; flood with 300% water; add 1.2% $NaHCO_3$ in 2 feeds at 30 min. interval; drum 2½ to 3 hr.; let stand overnight. Make certain that the pH is 5 to 5.5 throughout the thickness of the leather. Since the acidity of the leather increases in the vegetable retan, control of pH in the neutralization is important to guarantee a pH value not less than 3.5 and a difference figure on 10-fold dilution of at least 0.7; otherwise the leather will not meet (French) government specifications and Seal of Quality requirements. *Vegetable retan:* The neutralized leather is wrung under high pressure to reduce the water content

to the minimum, weighed, and drummed for 2 hr. with 10% powdered bisulfited quebracho and 10% powdered "sweetened" chestnut, dissolved in an equal weight of water at 80°–85°C. This makes an extract of 25° Bé. which is absorbed completely. Pile overnight, and wring again. Retan again with 10% powdered bisulfited quebracho dissolved in an equal weight of water, 2% talcum, and 1.5% fish oil. Mill for about 2 hr., until everything is taken up; then add 1% urea and 2.25% HCHO (30%, neutralized) and mill until completely taken up (about 1 hr.); add 0.4% oxalic acid; mill 1 hr.; pile overnight. The leather is then ready for the normal finishing operations. Shoulders processed as above fulfilled all requirements of the specifications except that mineral matter was considerably too high. This is attributed to mineral matter in the vegetable extracts used. It is claimed that these mineral salts, being chemically combined with the tannin, should not be considered deleterious, because of their different behavior compared to epsom salts. T.D.B.

Leather Manufacture and Instrumentation. F. E. Humphries. *Trans. Soc. Instrument Technol.*, **10**, No. 1, 1–12 (1958).—A general description of leather manufacture and control methods practiced in modern tanneries. H.B.M.

The Isoionic and Isoelectric Point of Acid-processed Gelatin from Insoluble and Citrate-extracted Collagen. D. S. Jackson and A. Neuberger. *Biochim. et Biophys. Acta*, **26**, 638–39 (1957) (in English); *Chem. Abstr.*, **52**, 5508*c*.

Nonaqueous Titration Technique. I. A Method for the Determination of Salts Present in Vegetable Tan Liquors. Y. Nayudamma and D. Ramaswamy. *Bull. Central Leather Research Inst., Madras*, **4**, 201–13 (1958).—This method is based on the fact that when the salts of weak acids are dissolved in nonaqueous solvents, the acid strength of the anion is so far suppressed that the cation can be titrated like a base. The procedure is as follows: Weigh 0.05 to 0.15g. of solid tanning extract, or 1 ml. of an extract solution or a spent tan liquor, and dissolve in 40 ml. of a 1:1 mixture of ethylene glycol and *n*-butyl alcohol. If desired, the extract may be dissolved first in the ethylene glycol before adding the butanol, or the extract may first be dissolved in not over 1 ml. water. The solution is titrated potentiometrically with 0.1*N* HCl in the ethylene glycol - *n*- butanol mixture. The end point is the point of inflection of the pH titration curve. When this is indistinct, the end point may be detected more exactly by plotting (ΔpH/Δml. acid) against pH. This plot gives a peak (sometimes 2) corresponding to the end point. A blank on the solvent is deducted. Preliminary tests with known salts showed that the method gave good results with sodium tannate, gallate, lactate, acetate, and formate. Less pronounced inflections were obtained with soduim oxalate, citrate, tartrate, and phosphate. The (ΔpH/Δml.) vs. pH plot for Na oxalate gave 2 peaks, but those for citrate, tartrate, and phosphate gave only one, corresponding to complete titration. Results are reported as meq. salt per 100 g. exract, or, in the case of extract solutions and liquors, as meq. salt per 100 ml. of 100 barkometer solution. Tests with white valem extract, which is high in salt, gave

the same salt content (254–258 meq/100 g.) for different sample weights up to 0.16 g., but a lower salt content for a larger sample. The presence of 2.5% water (1 ml. in 40) had a negligible effect, but the accuracy of the method declined rapidly at higher water contents. Results obtained for 7 extracts are given below, together with results obtained by titrating in aqueous solution to pH 2 (Atkin and Burton, abstracted *JALCA*, **44,** 888 [1949]):

	Meq. salt per 100 g.	
Extract	Nonaqueous titration (0.1 g. sample)	Aqueous titration to pH 2
Wattle	31	44
Quebracho	33	36
Terminalla arjuna	34; 68 *	67
Chestnut	15	16
Myrobalans	57	55
White valem	94; 254 *	241
Dhawa	135; 224 *	191

The asterisks mean that the (ΔpH/Δml.) vs. pH curves have 2 peaks, indicating that these extracts contain salts of 2 groups of acids that differ sharply in strength. Agreement between the nonaqueous titration method and the aqueous titration to pH 2 is quite good. Comparison with the ion exchange method (Okell, abstracted *JALCA*, **47,** 199 [1952]) and the sulfated ash method shows that the nonaqueous titration determines salts of weak acids only. The method has been checked by adding known amounts of salts to tanning extracts and has been shown to be applicable to tan liquors. The results are reproducible within about 3 to 5%. H.B.M.

Computers, Mathematics and Statistics. A. Rose, R. F. Sweeny, R. C. Johnson, W. F. Burggrabe, and R. L. Heiny. *Ind. Eng. Chem.*, **50,** 512–19 (1958).—A review of work published in 1957, with an extensive bibliography. H.B.M.

Drying. E. Bagnoli. *Ind. Eng. Chem.*, **50,** 435–37 (1958).—A review of work published in 1956–57 with 41 references. H.B.M.

Chelatometry. XVI. The Complexometric Determination of Iron and Aluminum in the Presence of Each Other. P. Wehber. *Z. Anal. Chem.*, **158,** 321–39 (1957); *Chem. Abstr.*, **52,** 4398*h*.

Physical Transitions in Collagen and Gelatin. R. R. Garrett. *Univ. Microfilms* (Ann Arbor, Mich.) *Publ.*, No. 24761, 133 pp.; *Dissertation Abstr.*, **17,** 2838–39 (1957); *Chem. Abstr.*, **52,** 4727*g*.

Wet Heating of Collagen. N. N. Stefanovich and A. N. Mikhailov. *Kolloid Zhur.*, **19,** 741–46 (1957); *Chem. Abstr.*, **52,** 5014*c*.

The Use of Disinfectants in Bringing Frigorifico Hides. M. Dempsey. *J. Soc. Leather Trades' Chemists*, **42**, 39–58 (1958).—In an effort to establish means of preventing "red heat" on brined hides during storage a commercial-scale test was conducted. Comparison was made of the effectiveness of adding Na pentachlorphenate and Na_2SiF_6 to the brine. Hides which did not contain either of these disinfectants were poorest after storage. Hides treated with Na_2SiF_6 were better, but even 1% silicofluoride was not enough to allow safe prolonged storage once "red heat" had begun. Best results were obtained with 0.2% Na pentachlorphenate. The hides were tanned at two sole leather tanneries, and their condition through the two yards and as finished leather is described. The disinfectants had no adverse effect on the leather although the flesh side of the hides brined with pentachlorphenate had a rather dingy color. Deterioration of hide where "red heat" developed was confirmed by volatile nitrogen determinations and by microscopic observation. J.M.C.

The Masking of Zirconium Sulfate Solutions. T. S. Ranganathan and R. Reed. *J. Soc. Leather Trades' Chemists*, **42**, 59–62 (1958).—Means of controlling the precipitation of Zr during neutralization of tanning liquors were studied by adding various organic reagents to basic Zr sulfate solutions. Of the reagents tested only acetate, formate, lactate, glycollate, gluconate, and citrate showed any masking properties. Acetate and formate did not greatly hinder the precipitation and are not regarded as useful masking agents. The remaining 4 reagents which were effective were Na salts of hydroxy carboxylic acids. J.M.C.

Studies in Calcification. The Reaction of Some Hard- and Soft-Tissue Collagens with 1-Fluoro-2:4-Dinitrobenzene. C. C. Solomons and J. T. Irving. *Biochem. J.*, **68**, 499–503 (1958).—Collagen samples prepared with a minimum of chemical treatment were obtained from ox tendon, hide, bone, and dentine, and from rat-tail tendon and human dentine. In general, demineralization of bone and dentine was effected by reaction with ethylenediamine tetraacetic acid. Treatment with fluorodinitrobenzene (FDNB) for 36 hr. was sufficient for dinitrophenylation of all tissues studied. Collagens of soft and of demineralized hard tissues could be distinguished from each other on the basis of the reactivity of their ε-lysyl and hydroxylsyl amino groups with FDNB. In intact soft-tissue collagen about two-thirds of the ε-amino groups reacted, but the concentration of reactive ε-amino groups of hard-tissue collagens increased from very low levels almost to theoretical at complete decalcification. J.M.C.

Self-vulcanizing Shoe Sole Adhesives. Hans. Herfeld. *Ges. Abhandl. Deut. Lederinsts. Freiberg/Sa.*, **No. 12**, 91–102 (1955).—Shearing strength, adhesive strength, and stability to flexure were measured for rubber-rubber, rubber-leather, and leather-leather joints. A rubber adhesive gave better joints than nitrocellulose for rubber-rubber and rubber-leather, but was not satisfactory for leather-leather joints. Poorer joints were produced when natural rubber in the adhesive was replaced by Buna S. Effects of variations in the formulation of the adhesive are described. H.B.M.

Chemicals from Wood. C. Placek, B. J. Buhmann, and R. T. Galganski. *Ind. Eng. Chem.*, **50,** 569–76 (1958).—A description of the processes employed by the Marathon Corporation for utilizing waste sulfite liquor. H.B.M.

Molecular Configuration of Gelatin. A. Veis and J. Cohen. *J. Polymer Sci.*, **26,** 113–16 (1957); *Chem. Abstr.*, **52,** 5936*h*.

Collagen. The Action of a Human Umbilical Cord Extract (Rich in Mucopolysaccharides) on Solutions of Collagen A. A. Delaunay, S. Bazin, R. Fauve, and M. Hénon. *Rev. Franc. Etudes Clin. et Biol.*, **1,** 165–73 (1956); *Chem. Abstr.*, **52,** 6432*d*.

Elastin, a Metabolically Active Lipoprotein. F. S. Labella. *Nature*, **180,** 1360–61 (1957); *Chem. Abstr.*, **52,** 6455*f*.

Rapid Assay of Sulfated Fatty Matters in Used Fat Liquors. J. Pore and B. Poirier. *Rev. Tech. Inds. Cuir.*, **50,** 27 (1958).—In some cases it is important to the tanner to know the amount of fat taken up by the leather during the fatliquor procedure. The classical methods of solvent extraction of the used bath after acid hydrolysis do not yield immediate results. The possibility of correcting a faulty absorption while the leather is still in process depends on the speed of the analytical method. By modification of a procedure described by Wachsmuth and Robyns, fatty matters are determined by a volumetric method and the volume converted into weight by use of a conversion factor. The analysis is performed in a 100-ml. measuring flask with the neck graduated in 0.1 ml above the 100-ml. mark. The flask is filled to the 100-ml. mark with the filtered emulsion to be analyzed, 1 ml. isoamyl alcohol is added to facilitate separation of the layers, 5–6 ml. conc. H_2SO_4 are added, and the flask is stoppered and agitated for one minute. The unstoppered flask is then kept for 10–15 minutes in a boiling water bath, cooled to room temperature, and the volume of the fatty matter collected in the neck of the flask is read. The same procedure may be used for sulfated oils by weighing an aliquot into the flask, dissolving or emulsifying it in water, and proceeding as above after bringing the volume up to 100 ml. A variety of fatliquors and commercial products were analyzed by the conventional as well as by the rapid method and the results compared by application of the empirically determined conversion factor. T.D.B.

The Degradation of Collagen. I. A Method for the Characterization of Native Collagen. A. Veis and J. Cohen. *J. Am. Chem. Soc.*, **76,** 2476–78 (1954).—Fresh steerhide was cut into strips, washed with cold water, extracted twice with cold 10% NaCl and once with ether, and swollen with cold 2% acetic acid. The flesh and grain layers were split off, and the resulting corium layer was deplumped with Na_2HPO_4, reswollen with 2% acetic acid, deplumped with Na_2HPO_4, washed with dilute NH_4OH, and dialyzed against cold, running distilled water. The resulting pieces were lyophilized, i.e., frozen and dried over

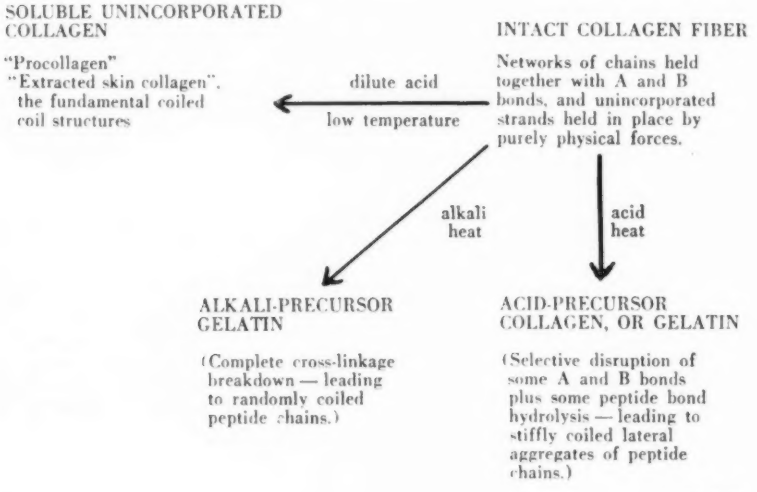
P_2O_5 in vacuum. Portions of the product were heated with 10 parts of water at 60°C. for 1, 2, 3, and 16 hr., filtered, washed, frozen and dried over P_2O_5. Up to 20% of the collagen was solubilized, but the remaining pieces looked like the original pieces. Portions of untreated and partially degraded collagens were treated with various concentrations of Orange G at pH 2.2, and of Safranin O at pH 11.5. Nonabsorbed dye was determined spectrophotometrically. Orange G at sufficiently high concentration binds with every cationic group of the collagen, degraded or not, but at lower concentrations the amount bound increases with the time that the collagen was heated. Safranine O, on the other hand, can combine with only about half the anionic groups of undegraded collagen, regardless of the dye concentration. Preheating the collagen makes the remaining anionic groups available as bonding sites. **II. The Solubilization Process in the Acid pH Range.** *Ibid.*, **77**, 2364–68 (1955).—Portions of collagen were heated at 60°C. for 1 to 4 hr. at pH 5.75 (isoionic), 4, 3, 2.5, and 2. The fractions solubilized were the same at pH 4 to 5.75 and increased progressively as pH was lowered below 4. The fractions solubilized appeared to approach limiting values at pH 2.5 and higher but not at pH 2. The nitrogen content of the insoluble residue decreased and that of the solubilized collagen increased as the fraction of the total collagen that was solubilized increased. The tentative interpretation of these facts is that high-temperature extraction of collagen involves primarily the separation of segments of a fairly uniform composition at a given value of pH and of fraction solubilized. Dye-binding experiments with the insoluble residues showed increased availability, but no increase in total number, of binding sites. Acid-base titration curves were run on the insoluble residues obtained at pH 5 (2 hr.) and pH 2 (4 hr), and on the corresponding solubilized fractions. These curves indicate that the dissolution process brings about an increase in effective pK value of some titrable groups. Fewer than 1 millimole of new free terminal amino groups per 100 g. of residual protein are created by the dissolution. If the solubilization process involves hydrolysis of peptide bonds, the soluble fragments must have molecular weights above 100,000. The difference in nitrogen content between the solubilized fractions and the residues suggests that collagen may consist of several related proteins of not quite identical amino acid composition. **III. Characterization of Soluble Products of Mild Acid Degradation.** A. Veis, D. N. Eggenberger, and J. Cohen. *Ibid.*, **77**, 2368–74 (1955).—Two soluble collagens, M (mild) and D (drastic) were prepared by extracting purified solid collagen at 60°C. for 1 hr. at pH 5.75 and for 4 hr. at pH 2 respectively. The soluble extracts were filtered, freed from salts by dialysis, and lyophilized. Solutions of M and D reached equilibrium after 4 hr. at 40°C. These high-temperature acid extracts differed notably from the low-temperature acid-soluble collagen in being stable at 40°C., while the low-temperature acid-soluble collagen hydrolyzes rapidly at 40°C. Soluble collagens M and D were fractionated by coacervation with alcohol, and with sodium dodecyl sulfate and NaCl. M and D responded very differently to the alcohol fractionation; less than 25% of D could be recovered as insolubilized fractions. M and D were separated into 5 fractions by sodium dodecyl sulfate – NaCl. Light-scattering and viscosity measurements were made on the unfractionated M and D, and on Fractions I and IV from each. The results of these measurements indicate great heterodispersity in all cases, with molecular weights ranging from 20 million down to about 1 million — D having the lower average molecular weights. The larger molecules appear to be stiff coils of 3 laterally aggregated, fundamental helix structures; the smaller molecules

(molecular weight 1 to 2 million) are stiffer, possibly segments of dissociated larger chains. They differ fundamentally from low-temperature acid-soluble collagen, which exists as randomly coiled single polypeptide chains, and from collagen (or gelatin) solubilized with alkali. H.B.M.

The Role of Cross Linkages in the Solubilization of Insoluble Collagen. A. Veis and J. Cohen. *J. Phys. Chem.*, **62**, 459–62 (1958); cf. preceding abstract.—An attempt was made to differentiate between the effects of H-bond and interchain-covalent bond rupture in the solubilization of collagen and the resulting distribution of molecular species. This was done by extracting collagen for 1 hr. at pH 5.75 at temperatures from 35° to 80°C., either in water or 2*M* KCNS, which breaks H-bonds. Extractions were also made in other H-bond-breaking solutions (urea, dimethyl formamide, phenol) but the results in these systems were intermediate between those in water and KCNS. Results in 2*M* NaCl were nearly the same as in water, indicating that ionic strength had little effect. Shrinkage temperatures were 55.8°–60.6°C. in water and 39.8°–43.2°C. in KCNS. The fraction solubilized in 1 hr. naturally increased with temperature and was much higher at any temperature in KCNS than in water. The extracts were fractionated with NaCl and alcohol. As found previously, the collagens that were solubilized under more drastic conditions contained more of the fractions of higher molecular weight and fewer of those of lower molecular weight. Entropy ($\triangle$ S), heat, and free energy of activation of the solubilization process were calculated from rates of solution at the different temperatures. The plots of fraction solubilized against temperature show sharp discontinuities both for the water and the KCNS system, but these do not coincide with T_s; they occur at higher temperatures than T_s. Also $\triangle$ S shows a sharp discontinuity at a temperature above T_s, both in water and in KCNS. The value of $\triangle$ S in water at temperatures below T_s is positive but much lower than $\triangle$ S of shrinkage, while in water at temperatures above T_s, and in KCNS both above and below T_s, $\triangle$S is negative. These negative entropy values can be explained as being due to increased immobilization of solvent. Disruption of H-bonds by heat or by KCNS makes hydration easier but is not the key process in solubilization. The discontinuity in $\triangle$ S corresponds to a sharp increase in solubilization of fractions of higher molecular weight. It is concluded that collagen "molecules" of different sizes exist in the intact structure, integrated into the fiber structure with different cross linkages or different cross-linkage densities. There appears to be no single homogeneous proto-fibrillar element. The polypeptide chains in collagen are not identical in amino acid composition (or sequence). Interchain linkages other than H-bonds are primarily responsible for the insolubility of collagen. H.B.M.

A Nonrandom Disaggregation of Intact Skin Collagen. A. Veis and J. Cohen. *J. Am. Chem. Soc.*, **78**, 6238–44 (1956); cf. preceding abstract.—Separate samples of collagen were extracted at 60°C. for 1 hr. at pH 5.75, 4, 3, and 2, and for 4 hr. at pH 2. Another sample was subjected to *successive* extractions at these pH values. A similar series of extractions was done at 80°C. and pH 5.75, 4, and 3. The percentage of the protein that was solubilized in each extraction was determined. Each extract was fractionated with NaCl plus alcohol. Molecular weights of the extracts, and of some of the fractions of those

extracts, were studied by light-scattering and viscosity measurements. From the data obtained it is concluded that intact skin collagen is, in some sense, a heterogeneous structure. The data are more in accord with the second of Ames's alternate hypothetical structures for collagen (*J. Soc. Chem. Ind.*, **66**, 279 [1947]), namely, that the intact fibrous structure is composed of segments which may vary in the degrees to which they are integrated into a larger macrostructure. By "integration" is meant the presence of interpeptide chain cross links, which may vary in density and/or type. It is inferred that 2 types of cross links exist in collagen, a primary set of acid-labile transverse bonds (A) and a second type (B) more stable towards acid and present in lower concentration than A. The heterogeneity of intact collagen can thus be ascribed to the presence of relatively few acid-stable B bonds, not necessarily distributed randomly throughout the structure. The collagen fibril is pictured as composed of segments varying in length and cross-section area due to differences in the cross-link distribution and the lateral ordering of polypeptide chains. The segments are chain structures held together by sets of acid-stable (B) bonds. These segments contain, and are held in the gross structure by, the acid-labile A bonds and physical forces. The disaggregation of collagen consists of various combination of A- and B-bond breakage and disruption of phyical forces to give "procollagen", acid-precursor collagen (gelatin), or alkali-precursor gelatin, as illustrated in the following schematic outline:


SOLUBLE UNINCORPORATED COLLAGEN
"Procollagen"
"Extracted skin collagen", the fundamental coiled coil structures
dilute acid
low temperature
INTACT COLLAGEN FIBER
Networks of chains held together with A and B bonds, and unincorporated strands held in place by purely physical forces.
alkali heat
acid heat
ALKALI-PRECURSOR GELATIN
(Complete cross-linkage breakdown — leading to randomly coiled peptide chains.)
ACID-PRECURSOR COLLAGEN, OR GELATIN
(Selective disruption of some A and B bonds plus some peptide bond hydrolysis — leading to stiffly coiled lateral aggregates of peptide chains.)

H.B.M.

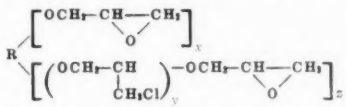
Ion Exchange Chromatography of Amino Acids. Effect of Resin Particle Size on Column Performance. P. B. Hamilton. *Anal. Chem.*, **30**, 914–19 (1958).—A back-washing technique was devised for grading spherical or irregularly shaped particles of sulfonated polystyrene cationic resins, approxi-

mately 8% cross-linked, into classes with diameters of 20, 25, 30, etc. microns, having 60 to 75% of the particles within ± 3 microns of the average diameter of the class, and a diameter range of approximately ± 7 microns. Martin's (or accidental) diameter, applicable to spheres and irregular particles alike, is proposed as a designation of particle size in preference to the less precise sieve number. Column performance in the separation of amino acids by ion exchange chromatography at the rapid flow rate of 39.3 ml. per sq. cm. of resin bed per hr. was optimum with spherical particles of 25-micron or irregular particles of 30-micron diameter. A range of 20 to 40 microns of pulverized resin was equally serviceable, if size distribution was uniform throughout the range.

Author's abstract

PATENTS

Tanning Agent and Process. U. S. Pat. 2,829,943. Edward M. Filachione and Edward H. Harris, Jr., Philadelphia, Pa., assignors to the United States of America as represented by the Secretary of Agriculture. Appl. Jan. 24, 1956. (Granted under Title 35, U. S. Code (1952), sec. 266).—1. A process of tanning hides comprising applying to the hides a tanning composition containing as the tanning agent a glycerol polyglycidyl ether corresponding to the formula



wherein R is a glyceryl residue, x is 1 to 2, y is 1 to 2, z is 1 to 2, and $x+z$ is 2 to 3 until the hides are tanned.

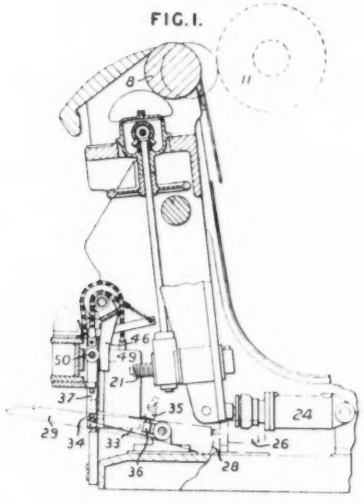
Method of Degreasing Wool and Recovery of Wool Grease. U. S. Pat. 2,833,798. Daniel P. Norman, Ipswich, Mass., assignor to Pacific Mills, Lawrence, Mass., a corporation of Massachusetts. Appl. Nov. 2, 1955.—1. The process for the removal of suint and wool grease from raw wool which comprises leaching the raw wool containing wool grease and suint with a mixture of methylene chloride and a liquid selected from the class consisting of water and polar organic liquids soluble in methylene chloride, to extract wool grease and suint therefrom, the amount of said liquid being less than 35% by weight on the wool.

Waterproofing Paper, Textiles, and Leather. Brit. Pat. 788,539. Svenska Oljeslageri Aktie-Bolaget. Appl. June 24, 1954.—Paper, textiles and leather are rendered hydrophilic by impregnation with aqueous solutions of Werner-type Chromium-III compounds containing inorganic acid radicals and organic carboxylic acid radicals (e.g. radicals of higher fatty acids or resin acids) wherein the stoichiometrical proportions between the chromium and the organic car-

boxylic acid radicals is 1 : 1 or 2 : 1. (For preparation, *see* Group IV (b)). In examples: (1) a dilute aqueous solution of a complex obtained by heating together potassium hydroxide, ethanol, sodium stearate and chromium-III chloride is used for impregnating paper, textiles and leather whereafter the impregnated materials are dried and heated to 115° C.; leather when treated immediately after tanning and whilst still wet absorbs the chromium complex from solution at *p*H 2.9–3.5 and it is unnecessary to heat after drying; (8) a dilute solution of a complex obtained from stearic acid, chromium-III chloride, isopropyl alcohol and sodium bichromate is used for impregnating leather.

Hide-Treating Machines. Brit. Pat. 788,474. Turner Machinery, Ltd., [formerly Turner Tanning Machinery Co., Ltd.], and Hall C. Appl. Sept. 3, 1956.—A machine for shaving or otherwise working hides and skins, in which a work support is moved by treadle-operated means to and from a rotary tool, is provided with at least one stop-projection co-operating with an abutment yieldable under the control of the operator and preferably by the treadle, so as to arrest the approach stroke of the support. As shown in Fig. 1, as a result of the shape of the cam edge 28, depression of the treadle 29 progressively opens the control valve 26 of the hydraulic cylinder 24 until about midway through the treadle swing the valve is fully open. The carrier is swung hydraulically to bring the work support roller 8 towards the shaver roller 11 until the screwed stop projection 21 meets the face 49 of the abutment 46. A lost-motion coupling is provided between the treadle and the yoke 37 by a stud 35 which only contacts a lug 36 to commence movement of the arm 33 connected by the pin 34 to the yoke 37 when the treadle has been depressed sufficiently to open the valve fully. Further depression of the treadle lowers the roller 50 gradually

FIG. 1.



to retract abutment face 49 and to allow the carrier to swing a little further in the approach direction so that the hide is gradually presented to the shave roller 11.

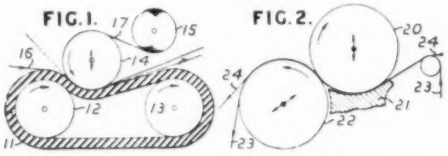
Waterproofing. Brit. Pat. 790,038. Midland Silicones, Ltd. Appl. Jan. 31, 1956.—Fabrics, e.g. of cotton, wool, linen, ramie, silk, suede, leather, camel's hair, rayon, nylon, vinylidene chloride-acrylonitrile, acrylonitrile, ethylene glycol-terephthalic acid ester, paper, asbestos or glass, are rendered water-repellent by applying thereto, e.g. by spraying, dipping, flooding or brushing, an aqueous emulsion comprising: (1) from 0.5 to 20 per cent by weight of a hydrocarbon solvent-soluble polysiloxane having an average of from 0.05 to 1 silicon-bonded hydrogen atom per Si atom, the remaining valencies of the Si atoms being satisfied by oxygen atoms and monovalent hydrocarbon radicals, there being an average of from 1.75 to 2.5 total hydrocarbon radicals and hydrogen atoms per Si atom, and (2) from 80 to 99.5 per cent by weight of water and a water-soluble alkanolamine titanate or zirconate (*see* Group IV (b)) or a hydrolysate thereof, the amount of the titanate or zirconate being such that there is in the emulsion from 0.5 to 15 per cent by weight of metal based on the weight of the polysiloxane. The fabric is then dried and the siloxane cured, e.g. by heating from 5 to 20 minutes at 100° to 150° C. Emulsifying agents, preferably of the non-ionic type such as glycerol mono-oleate, glycerol stearate and polyvinyl alcohol, may be added, as well as aminoplasts such as urea- and melamine-formaldehyde resins. Examples are given.

Unhairing and Bating. Brit. Pat. 790,237. Soc. Des Laboratoires Labaz. Appl. May 11, 1955.—Skins are bated and/or unhaired by bringing into contact, as by immersion, painting, spraying or fulling, with a culture of a variety of Streptomyces having caseinolytic, keratinolytic and/or unhairing activity, or a filtrate thereof. The culture or filtrate may be concentrated in vacuo before use, and may be used in a neutral or alkaline medium. Specified Streptomyces are S. albus (G), S. antibioticus, S. aureus and S. griseus and unnamed varieties derived from putrescent sheepskin, air, earth and pond-mud. Suitable tests by which the presence and magnitude of the lytic activities may be determined are described. Some of the Streptomyces varieties used may also have antibiotic activity due to secretion of actinomycin or actinomycetin. An exemplified culture contains peptone and $NaNO_3$, KCl, K_2HPO_4, $MgSO_4$ and $Co(NO_3)_2$.

Leather, Paper, and Textiles Containing Synthetic Resins &c.; Coated Fabrics. Brit. Pat. 790,585. Rohm & Haas Co. Appl. Mar. 13, 1956.—Homopolymers of acrylyl and methacrylyl dicyandiamides, copolymers of the latter monomers with one or more other monoethylenically unsaturated compounds and reaction products of such polymers with hydrogen sulphide, formaldehyde, acetaldehyde, glyoxal and urea-formaldehyde, thiourea-formaldehyde and melamine-formaldehyde preliminary condensation products are used alone or in conjunction with urea-formaldehyde or melamine-formaldehyde condensates for coating or impregnating leather, paper and wool, cotton and rayon textiles or for imparting wet strength to paper. In examples: (1) methacrylyl dicyandiamide and N - (β - methacrylamidoethyl) - N : N' - ethylene-urea are

copolymerized in aqueous dimethyl-formamide using dimethyl azodiisobutyrate as a catalyst, the product is heated with aqueous formaldehyde and added to unbleached kraft pulp which is formed into paper sheets; (2) red iron oxide is dispersed in water containing sodium lauryl sulphate and *tert.*-octylphenoxy-polyethoxyethanol, methyl cellulose and an aqueous emulsion of an ethyl acrylate-acrylyl dicyandiamide copolymer is added thereto and the resulting pigmented dispersion is swabbed on to full grain upholstery leather and dried at 50° C.

Ornamenting Leather and Paper; Finishing Fabrics. Brit. Pat. 791,553. Bradford Dyers Association, Ltd., and Bleachers Association, Ltd. Appl. Feb. 6, 1956.—A compressively shrinkable sheet material is ornamented by compressive-



ly shrinking it in contact wth a second sheet material which buckles, the buckles so formed being transferred to the first material. The compressively shrinkable materials include fabrics, paper, leather, parchment, regenerated cellulose and plastic sheets, and coated textiles such as book-cloth and imitation leather cloth. The second material may be of paper, cardboard, a plastic, thin sheet metal, a fabric stiffened by a resin, starch or other agent, or a fabric so woven that it will not readily shrink, but tends to buckle if compressively shrunk alone. The compressively shrinkable material may be of nylon, polyethylene terephthalate or other thermoplastic which is heated during treatment to maintain the ornamenation, or it may be pretreated with a temporary plasticiser or an agent which will set by heat applied during or after the ornamentation, or by a chemical reagent applied after the ornamentation. The compressively shrinkable material may be disposed between two sheets of material which buckles during treatment, and compressive shrinking may be applied discontinuously over the surface. As shown in Fig. 1, the compressively shrinkable material 16 is fed on to a thick rubber or like belt 11 passing around rollers 12 and 13 at a point in advance of that at which the belt begins to contract, and the second sheet material 17 is fed from a roll 15 into the nip between the belt 11 and a roller 14. In the apparatus of Fig. 2, the compressively shrinkable material 23 and the second material 24 are fed into an inlet nip between a roller 20 and an electrically heated bed plate 21, and emerge from an outlet nip formed by the roller 20 and a roller 22, the roller 20, which has a hard rubber surface, rotating faster than the roller 22, which has a soft rubber surface. Discontinuous apparatus, e.g. a press, one face of which may be caused to contract in length, may also be used.

Making Leather. Brit. Pat. 790,933. Boehme Fett-Chemie Ges. Appl. Aug. 2, 1955.—Tanned or untanned leather or skins are tanned, retanned or filled

by contacting said skin in any order with aqueous solutions of a water-soluble salt of a polymeric inorganic or organic acid and of a water-soluble cationic resin. Polymeric acids are defined as silicic, phosphoric, polyacrylic and polymethacrylic acids; alkali metal or ammonium salts or salts of organic bases are suitable. When using waterglass solutions, the *p*H is usually first adjusted with acetic acid. Suitable resinous substances are those containing amino or imino groups which have been reacted with oxo compounds. Specific nitrogen compounds referred to are urea, thiourea, methylenediurea, cyanamide, guanamine, alkylguanidines, dicyandiamide, dicyandiamidine, guanidine, ammeline, ammelide, and melamine. Suitable oxo compounds are formaldehyde, acetaldehyde (and compounds yielding them), acrolein, furfural and methylethylketone. Further basic components, e.g. polyamino compounds such as polyalkylenepolyamines, e.g. triethylenetetramine, tetraethylenepentamine, hydroxyalkylamines such as dimthylaminoethanol, diethanolamine, oxazolidine, and polyphenyldiguanides may be subsequently condensed with the initial condensate. The process may be preceded or followed by tanning with formaldehyde, vegetable, synthetic, mineral tanning substances or tanning substances having an aliphatic radical with at least 6 carbon atoms, examples of which are chrome, aluminium and zirconium salts, and train oil, alkyl sulphates, alkyl sulphonates, alkyl benzene sulphonates and the corresponding sulphochlorides. An anion-active tanning agent having an aliphatic radical with at least 6 carbon atoms, e.g. an alkyl sulphate or alkyl benzene sulphonate, may partially replace the polymeric acid salt. In examples: (1) a chrome-tanned hide is treated with an acidified waterglass solution followed or preceded by a solution of a dicyandiamide/guanidine hydrochloride/formaldehyde condensation product in the same or a separate bath. (2) Delimed and bated pelts are first treated with the condensation product in (1), or a melamine/formaldehyde condensate, followed by waterglass; if desired the so-produced leather may be chrome-retanned. (3) Delimed and bated pelts are first treated with the condensation product in (1), or a dicyodiamide/formaldehyde condensate, then with sodium hexametaphosphate and finally retanned with a nitrogen-containing cationic condensate. (4) Delimed and bated pelts are pickled in a salt/sulphuric acid bath, a condensate as in (1) is added, the pelts are treated in a chrome bath and then first with sodium hexametaphosphate and finally with a further bath of the same condensate. (5) Chrome-tanned leather is neutralized and treated first with sodium hexametaphosphate (this could be replaced by sodium polymethacrylate) and then with a guanylurea hydrochloride/formaldehyde condensate.

Waterproofing Leather. Brit. Pat. 791,663. Boehme Fettchemie Ges. Appl. Dec. 1, 1955.—A process for the impregnation of leather to render it water-repellent comprises treating the leather with an organic metal complex obtained by reaction of a basic salt of a polyvalent metal with a derivative of an inorganic or organic acid having a basicity of at least three, which derivative contains a higher molecular weight aliphatic, cycloaliphatic, aliphatic-aromatic or aliphatic-cycloaliphatic residue. If the complex to be employed is an organic chromium complex, this may be prepared in situ by applying successively to the leather in either order a basic chromium salt of the derivative, which latter may be in the form of a water-soluble salt thereof such as an ammonium or alkali metal salt or a salt with an organic base. Specified inorganic acids whose derivatives may be used are boric acid, phosphoric acid and arsenic acid; specified organic acids

are citric, trimesic, trimellitic, mellitic, diadipic, ethane-tetra-acetic, sulphophthalic, hydroxynaphthalic, pyrogallic and pyrogallol-carboxylic acids, and also condensation products of oleic acid, linseed oil fatty acid and soya oil fatty acid with maleic acid. The last-named higher induction weight carboxylic acids may be utilized as such. Specified derivatives are acid dodecyl phosphate, acid octadecenyl phosphate, hexadecyl borate, octadecenyl arsenate, acid hexadecyl citrate, acid dodecyl sulphophthalate, the mononaphthenylamide of citric acid and the monooctadecylamide of citric acid. The application of the derivatives to leather when the complex is to be prepared in situ may be effected by dipping or brushing with aqueous dispersions or solutions. If the complex is to be prepared before treating the leather, specified derivatives which may be used are the mono- or di-octyl, mono- or di-dodecyl, mono- or di-oleyl esters of orthophosphoric acid, or mixtures of acid esters obtained by the action of phosphorus pentoxide on higher-molecular weight hydroxy compounds. Specified basic salts of polyvalent metals are basic chromium chloride, basic aluminium nitrate and basic titanium chloride. The basic metal salts may be reacted with the phosphoric acid esters in solution in methanol or ethanol, or in the molten state. The application of the complexes in solution to the leather may be effected by dipping, fulling, brushing, plushing or spraying. Examples disclose impregnating chrome-tanned leather with an aqueous solution of an ammonium salt of an acid dodecyl phosphate or an aqueous solution of an ammonium salt of a polycarboxylic acid obtained by reaction of linseed oil fatty acid and maleic acid; impregnating back-tanned leather with an aqueous solution of an ammonium salt of the mono-oleyl ester of citric acid followed by treatment with Cr_2O_3 in the form of a chrome-tanning substance; reacting basic chromium chloride with the monotetradecyl ester of phosphoric acid or with a mixture of alkyl esters of phosphoric acid (alkyl residues C_{12}—C_{18}) in methanol and impregnating leather with the resultant solution; reacting Cr_2O_3 with an ammonium salt of the mono-oleyl ester of citric acid or with an ammonium salt of an acid dodecyl phosphate, in water, and impregnating leather with the resultant solution; and reacting basic aluminium chloride with a mixture of alkyl esters of phosphoric acid (alkyl residues C_{12}—C_{18}) in methanol and then impregnating leather with a solution of the resultant complex in alcohol.

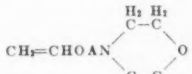
Fat-liquoring Leather. Brit. Pat. 788,657. Boehme Fettchemie Ges. Appl. June 25, 1954.—Chrome-tanned hides are treated with a fat-liquoring oil containing a masking agent which is an oil soluble partial ester or partial amide of a carboxylic acid containing 3 or more carboxyl groups, or phosphoric acid, with high molecular weight alcohols or amines, or a salt of said partial esters and partial amines. Acids specified are phosphoric, diadipic, citric, 1,2,3,4 butane-tetracarboxylic, trimesic, mellitic, nitrilo-tri-acetic, and ethylene diamine-tetra-acetic acids, and water-soluble salts of such esters and amides. Examples of masking agents are the monododecyl ester of orthophosphoric acid, the monooctadecyl ester of citric acid, the mono-dodecyl ester of nitrilo-triacetic acid and the mono- and di-dodecyl esters of ethylene diamine tetra-acetic acid. Normally 2 to 20 per cent of such agents are incorporated in the fat-liquoring oil. Examples relate to (1) a fat-liquoring oil composed of 40 parts of sulphonated sperm oil, 40 parts of un-sulphonated sperm oil, and 10 parts of mineral oil to which was added 10 parts of the sodium salt of the monododecyl ester of ortho-phosphoric acid. 1.2 to 3.5 per cent of this composition was employed in about 200—250 per cent of liquor for the fat-liquoring of chrome-

tanned upper leather. (2) a fat-liquoring oil similar to (1) above contained 15 per cent of the cyclohexyl amine salt of the mono-oleyl ester of ortho-phosphoric acid as a masking agent, and was applied in an amount of 4—6 per cent to chrome-tanned leather, the application being such that one-third of the fat-liquor oil was applied to the chrome-tanned and neutralized leather, followed by charging with 3—6 per cent of a vegetable tanning material, dyeing, and again fat-liquoring with the remainder of the fat-liquoring oil. (3) To the fat-liquoring oil of Examples (1) and (2) above 10 per cent of the ammonium salt of a mono-ester of citric acid was added in place of the masking agent mentioned. The fatty alcohol fraction used for the esterification consisted of coconut oil alcohols C_{12} to C_{18} chain length.

Waterproofing Leather. Brit. Pat. 788,741. Imperial Chemical Industries, Ltd., Appl. Dec. 9, 1955.—A process of treating leather comprises introducing into a leather in the presence of a water-soluble zirconium salt an organo-polysiloxane from an aqueous emulsion thereof containing a water-soluble soap and then treating the leather with a solution of a water-soluble aluminium salt. Specified soaps are ammonium oleate, potassium stearate or sodium palmitate. A specified polysiloxane is methyl polysiloxane. A specified zirconium salt is zirconium acetate. Specified aluminium salts are aluminium sulphate and potash alum. Examples describe the treatment of leather skins by drumming them with an aqueous emulsion of methyl polysiloxane containing zirconium acetate and a soap followed by treatment with a solution of aluminium sulphate or potash alum.

Method of Coating Leather with Polymers Containing Units of Acrylyl or Methacrylyl Dicyandiamide and the Resulting Article. U. S. Pat. 2,828,222. Benjamin B. Kine, Levittown, and Paul W. McWherter and Hugo A. Alps, Philadelphia, Pa., assignors to Rohm & Haas Company, Philadelphia, Pa., a corporation of Delaware. Appl. March 21, 1955.—18. The method of finishing leather comprising applying thereto an aqueous dispersion of a water-insoluble linear polymer of monoethylenically unsaturated molecules comprising 3% to 50% by weight of at least one monomeric compound selected from the group consisting of acrylyl and methacrylyl dicyandiamides, then drying the coated leather, applying thereto a lacquer, at least one of said dispersion and lacquer being pigmented, and drying the lacquer.

Method of Coating Leather with Polymers Containing Morpholino Groups and the Resulting Article. U. S. Pat. 2,828,223. Paul W. McWherter, Philadelphia, Benjamin B. Kine, Levittown, and Hugo A. Alps, Philadelphia, Pa., assignors to Rohm & Haas Company, Philadelphia, Pa., a corporation of Delaware. Appl. Mar. 21, 1955.—1. As an article of manufacture, a leather having a coating thereon comprising a water-insoluble linear polymer of monoethylenically unsaturated molecules comprising at least 3% by weight of a monomeric compound having the structure of the formula



where A is an alkylene group having 2 to 10 carbon atoms.

Method of Coating Leather with Polymers Containing Ureido Groups and the Resulting Article. U. S. Pat. 2,828,224. Hugo A. Alps and Paul W. McWherter, Philadelphia, and Benjamin B. Kine, Levittown, Pa., assignors to Rohm & Haas Company, Philadelphia, Pa., a corporation of Delaware. Appl. Mar. 21, 1955.—1. As an article of manufacture, a leather having a coating thereon comprising a water-insoluble linear polymer of monoethylenically unsaturated molecules comprising at least 3% by weight of a monomeric compound having the structure of formula

(I) $$CH_2{=}(R'')YANRCONHR^1$$

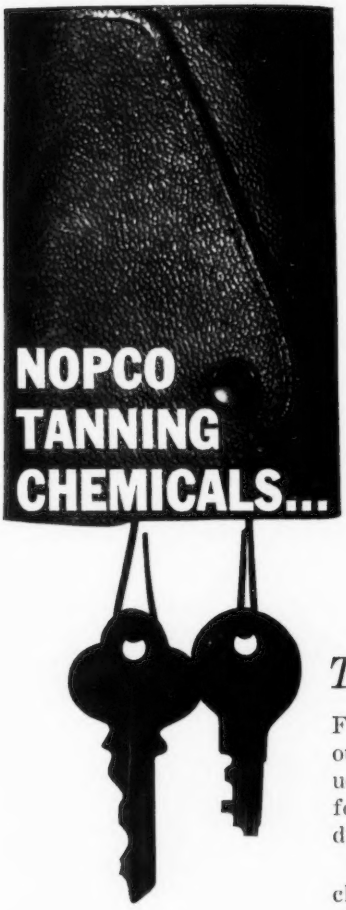
where R'' is selected from the group consisting of H and CH_3, Y is selected from the group consisting of O, —COO—, and —CONH—, A is selected from a class consisting of branched and straight chain alkylene groups having 2 to 4 carbon atoms between adjoining oxygen and nitrogen atoms and etheroxygen linked alkylene groups having 2 to 3 carbon atoms, and R and R^1 are each selected individually from the class consisting of hydrogen, lower aliphatic groups having 1 to 3 carbon atoms, and ether-linked lower aliphatic groups having 1 to 3 carbon atoms, except that R may form with R^1 a ring-closing alkylene group selected from the class consisting of ethylene, propylene, and trimethylene.

Method of Coating Leather with Polymers Containing Hydroxyl Groups and the Resulting Article. U. S. Pat. 2,828,220. Paul W. McWherter, Philadelphia, Benjamin B. Kine, Levittown, and Hugo A. Alps, Philadelphia, Pa., assignors to Rohm & Haas Company, Philadelphia, Pa., a corporation of Delaware. Appl. Mar. 21, 1955.—1. As an article of manufacture, a leather having a coating thereon comprising a water-insoluble linear polymer of monoethylenically unsaturated molecules comprising (1) 70 to 97% by weight of an alkyl ester of an acid selected from the group consisting of acrylic, methacrylic, and itaconic acids in which the alkyl group has 1 to 8 carbon atoms and (2) 3% to 30% by weight of a monomeric compound having the structure of formula

$$CH_2{=}CH—A—R—OH$$

where A is selected from the group consisting of —O— and —S—, and R is selected from the group consisting of straight and branched chain alkylene groups having from 2 to 10 carbon atoms, $—C_2H_4AC_2H_4—$, $—C_2H_4N(R^1)C_2H_4—$, and $—C_2H_4N(R^2)(R^3)(X)C_2H_4—$ where R^1 is selected from the group consisting of H, CH_3, C_2H_5, and C_2H_4OH and R^2 and R^3 are individually selected from methyl, ethyl, and hydroxyethyl ($—C_2H_4OH$) groups and X is a negative radical.

Method of Coating Leather with Polymers Containing Cyclic T-Amino Groups and the Resulting Article. U. S. Pat. 2,828,221. Paul W. McWherter, Philadelphia, Benjamin B. Kine, Levittown, and Hugo A. Alps, Philadelphia, Pa., assignors to Rohm & Haas Company, Philadelphia, Pa., a corporation of Delaware. Appl. Mar. 21, 1955.—18. The method of finishing leather comprising applying thereto an aqueous dispersion of a water-insoluble linear polymer of monoethylenically unsaturated molecules comprising 3% to 50% by weight of at least one monomeric compound selected from 4-vinylpridine and 2-methyl-5-vinylpyridine, then drying the coated leather, applying thereto a lacquer, at least one of said dispersion and lacquer being pigmented, and drying the lacquer.



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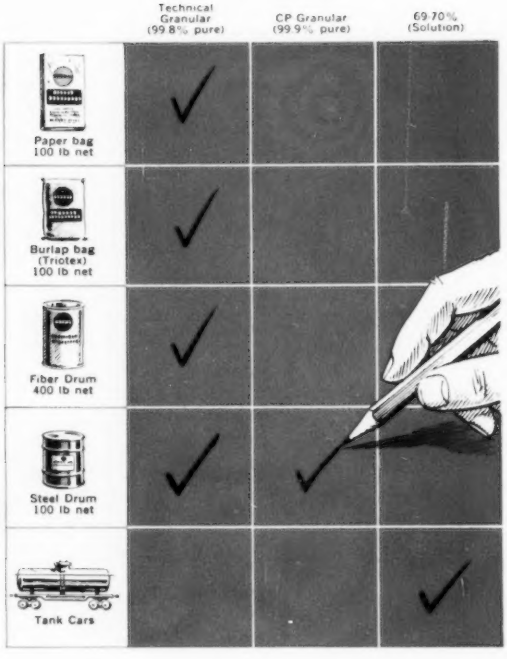
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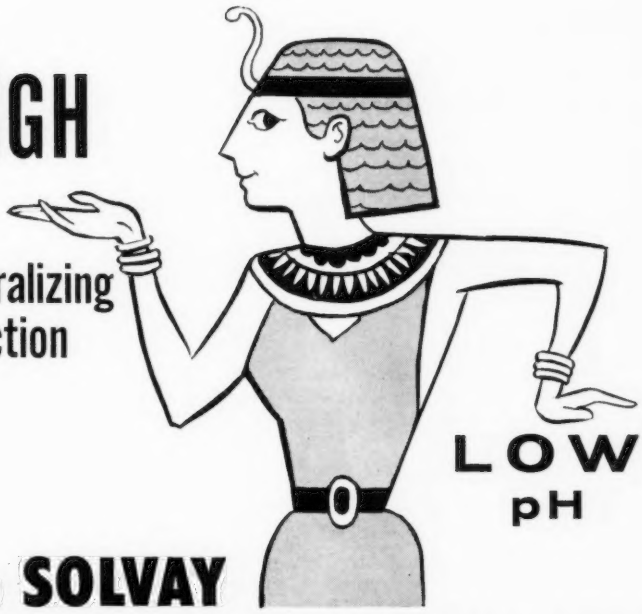

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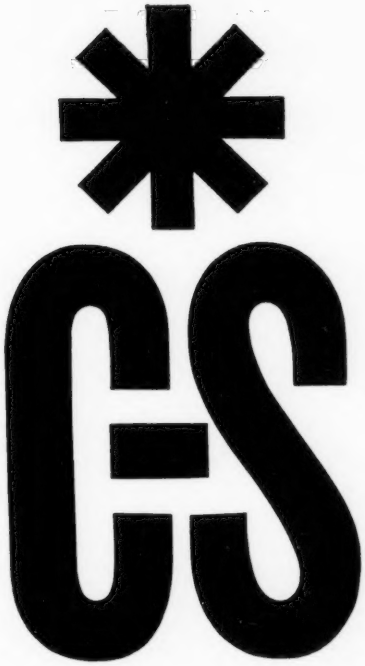

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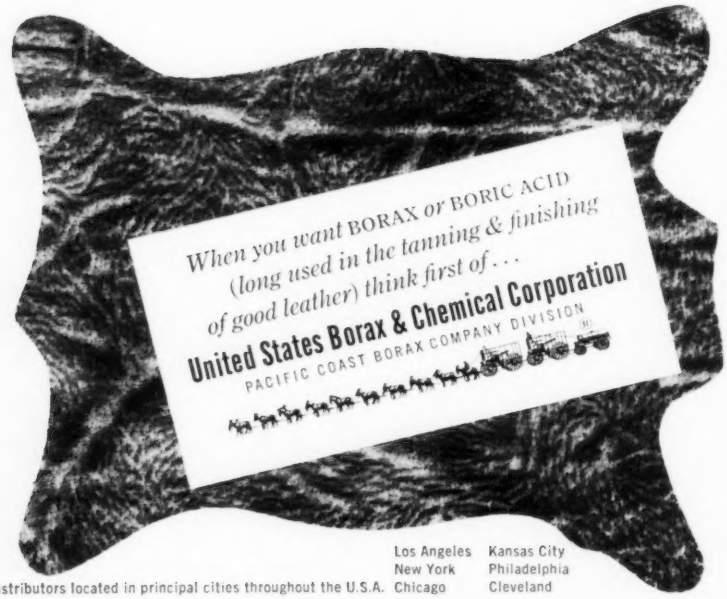
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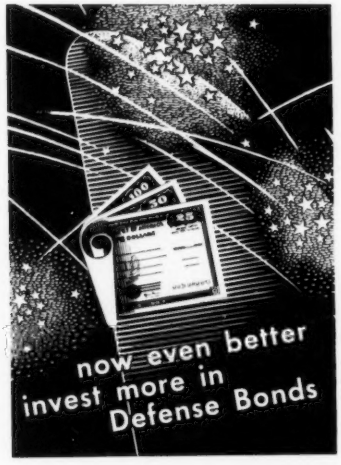

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They made a number of attempts to overcome this problem without success. Then they discovered Synova. By top fatliquoring with only a small percentage of Synova, they found they could get a level, beautifully cleared surface, properly conditioned to receive the transparent finish.

Tannery Run Upgraded

Now oil spotting has been eliminated in this tannery. Their product is far more uniform, their colors bright and level, their stock upgraded on the table.

Also they found that Synova can be used at temperatures as high as 130°F—an obvious advantage since it can be applied on top of the regular fatliquor at the same temperature. This eliminates chill floating and door changing. What's more, Synova has proved absolutely uniform, with long storage stability—and it has a clean, pleasant odor.

Recently this tannery began using Synova to clear the grain on softie type leathers also. Formerly these had too much surface feel because maximum amounts of fatliquor were used to get the desired degree of softness. But now this condition is eliminated by top fatliquoring with only 1% Synova.

Maybe YOU Can Profit Too

If your tannery produces aniline or sem-aniline finish grains, or softie and tropical leathers, it's just plain good business to see what modern Synova can do for you.

Try it for yourself. Let us send you a trial sample, without any obligation. Write or phone today.

It's time you switched to Synova!

SEABOARD CHEMICALS, INC.

Dept. 8C, 30 Foster St.

Salem, Massachusetts

In Canada: Tennant Union Ltd., Toronto

Overseas Export: Wolff International, Inc., Milwaukee, Wisconsin, U. S. A.

A new chemical is born— will it live?

Not more than one out of 75 new products survives the various screening tests and reaches the market. Evaluation in this minute tanning equipment is the first hurdle to be overcome.

CHEMTAN COMPANY
EXETER, NEW HAMPSHIRE

All our facilities are devoted exclusively to the tanning industry.